

In presenting the dissertation as a partial fulfillment of the requirements for an advanced degree from the Georgia Institute of Technology, I agree that the Library of the Institute shall make it available for inspection and circulation in accordance with its regulations governing materials of this type. I agree that permission to copy from, or to publish from, this dissertation may be granted by the professor under whose direction it was written, or, in his absence, by the Dean of the Graduate Division when such copying or publication is solely for scholarly purposes and does not involve potential financial gain. It is understood that any copying from, or publication of, this dissertation which involves potential financial gain will not be allowed without written permission.

3/17/65

b

MEASUREMENT OF THERMAL CONDUCTIVITY
DURING FREEZE-DRYING OF BEEF

A THESIS

Presented to

The Faculty of the Graduate Division

by

William M. Massey, Jr.

In Partial Fulfillment

of the Requirements for the Degree

Master of Science in Mechanical Engineering

Georgia Institute of Technology

March, 1966

MEASUREMENT OF THERMAL CONDUCTIVITY
DURING FREEZE-DRYING OF BEEF

Approved:

Chairman

Date approved by Chairman: 3/7/68

ACKNOWLEDGMENTS

The author is indebted to a number of people whose help and encouragement have made this work possible. First and especially he would like to express his appreciation to his advisor, Dr. J. E. Sunderland, whose instruction and advice were an invaluable aid throughout this work. Thanks are due to Dr. C. W. Gorton and Dr. L. J. Ybarrondo for their advice and service on the reading committee. Also, the writer appreciates the partial support from the Public Health Service Research Grants EF 00102-02 and EF 00102-03 from the Division of Environmental Food Protection. A special note of thanks is due to Dr. D. F. Dyer whose comments and advice were very helpful and to Mr. C. R. Bannister for the fabrication of the experimental apparatus.

The author would like to express his appreciation to his parents, Mr. and Mrs. W. M. Massey, Sr. whose guidance, encouragement and support have been invaluable to him in his educational work. Finally, sincere appreciation is due to his wife, Nita, and his daughter, Michelle, without whose love, encouragement and help this work would not have been possible.

TABLE OF CONTENTS

	Page
ACKNOWLEDGMENTS	ii
LIST OF TABLES	v
LIST OF ILLUSTRATIONS	vi
SUMMARY	viii
NOMENCLATURE	xi
CHAPTER	
I. INTRODUCTION	1
Statement of Intent	
Freeze-Drying	
Measurement of Thermal Conductivity	
Thermal Conductivities	
II. THEORETICAL ANALYSIS	10
Thermal Conductivity of Freeze-Dried Beef	
Quasi-Steady Energy Balance for Freeze-	
Drying Process	
Alternate Method for Conductivity	
Measurement	
III. EQUIPMENT AND INSTRUMENTATION	22
Equipment	
Instrumentation	
IV. EXPERIMENTAL PROCEDURE	28
Pretest Procedure	
Test Procedure	
Post Test Procedure	
Test Procedure for Alternate Method of	
Conductivity Measurement	
V. PRESENTATION AND DISCUSSION OF RESULTS	35
Calculation Procedure	

CHAPTER	Page
V. (Continued)	
Experimental Results	
Results for Alternate Conductivity Test	
Calculated Drying Times	
Experimental Errors	
VI. CONCLUSIONS AND RECOMMENDATIONS	64
APPENDICES	
A. TABULAR DATA FOR CONDUCTIVITY TESTS	66
B. DERIVATION OF DRYING TIME EQUATION	67
C. EQUIPMENT AND INSTRUMENT SPECIFICATIONS	71
LITERATURE CITED	73

LIST OF TABLES

Table	Page
1. Results of Thermal Conductivity Tests on Freeze-Dried Beef	41
2. Surface and Interface Temperatures for Conductivity Tests	44
3. Thermal Conductivities at 1 torr Chamber Pressure	48
4. Comparison of Predicted Drying Times with Experimental Drying Times	50
5. Comparison of Predicted Interface Temperatures and Experimental Interface Temperatures	60
6. Weight Loss for Test Runs at Each Chamber Pressure	66

LIST OF ILLUSTRATIONS

Figure	Page
1. Schematic of Freeze-Drying Model for Thermal Conductivity Analysis	13
2. Schematic of Test Sample for Special Thermal Conductivity Test with Ice Between Two Dry Beef Samples	20
3. Schematic Diagram of Freeze-Drying Test Equipment	23
4. Schematic of Prepared Sample for Thermal Conductivity Test	31
5. Plot for Determination of Thermal Conductivity, 0.2 torr and 0.5 torr	38
6. Plot for Determination of Thermal Conductivity, 0.7 torr and 1.0 torr	39
7. Plot for Determination of Thermal Conductivity, 2.0 torr and 3.0 torr	40
8. Thermal Conductivity of Freeze-Dried Beef versus Total Chamber Pressure (Heat of Sublimation = 1220 Btu/lb _m)	43
9. Comparison of Thermal Conductivity Results During Freeze-Drying and Steady-State Thermal Conductivities	46
10. Moisture Content versus Time, 1 torr	52
11. Moisture Content versus Time, 2 torr	53
12. Moisture Content versus Time, 3 torr	54
13. Interface Position versus Time, 1 torr	56
14. Interface Position versus Time, 2 torr	57
15. Cross-section of a Partially Dried Beef Sample, 1/2 inch Thick	62

LIST OF ILLUSTRATIONS (Continued)

Figure	Page
16. Cross-section of a Partially Dried Beef Sample, 1-1/2 inches Thick	62

SUMMARY

Thermal conductivities of freeze-dried foods are needed in order to predict drying rates. The objective of this thesis is to report measurements of the thermal conductivity of freeze-dried beef.

The conductivity of dry beef is given from data obtained during freeze-drying processes. At the start of the drying cycle, a dry layer forms over the surface of the beef. The location where dried and frozen portions meet is a distinct plane called the "interface." As drying continues, the interface recedes gradually into the sample. An energy balance is made on the dry layer by equating the heat conducted across the dried layer to the weight loss of the sublimating sample. The convective contribution to heat transfer due to the vapor flowing through the dry layer is also included in the heat balance. Transient effects are excluded in the analysis by considering only short time intervals. The analysis is thus termed "quasi-steady" as spatial properties are considered constant for short periods of time. The very slow process of freeze-drying makes the quasi-steady assumption valid.

In this work, beef samples are dried at pressures ranging from 0.2 to 4 torr. The results show that the conductivity increases with increasing pressure. For example, the conductivity at 1 torr is about 35 per cent greater than the conductivity at 0.2 torr. As the pressure increases above 1 torr, the rate of change of conductivity with pressure becomes smaller. The conductivity at 3 torr is only 3 per cent greater

than the conductivity at 1 torr. At a chamber pressure of 4 torr the beef apparently thawed and, although drying occurred, the process ceased to be sublimation dehydration.

The conductivities measured under drying conditions do not seem compatible with results obtained using conventional steady-state means on dry samples. Under steady-state conditions various gases are substituted in the void spaces in place of the actual gas mixture during freeze-drying. Therefore the conductivity data of this work are used in drying rate equations to compare theoretically predicted drying rates with experimentally observed results. In all cases the theoretical and experimental data compare very well.

An attempt is made to resolve the differences between conductivity results obtained during drying and steady-state measurements. The conductivity of previously dried beef, measured by sublimating a piece of ice between two dried beef samples is presented. In this test the water-vapor passes through the beef samples into the vacuum chamber. An energy balance similar to that used during freeze-drying allows calculation of the thermal conductivity of the beef. Although this procedure approximates both the steady-state procedure and the quasi-steady procedure, the results are inconclusive due most likely to thermal differences between water-vapor and beef juice-vapor.

The heat of sublimation of pure ice is used by other investigators to calculate the conductivity using experimental methods like those presented in this thesis. The same property data are used in this work. Also, a recently measured value of the heat of sublimation of frozen beef juices is used to calculate the conductivity. The difference between

the two calculated conductivities is significant, but it is shown that theoretical drying rates are unaffected since the appropriate heat of sublimation is also used in the drying rate equations.

This work indicates that measurement of thermal conductivities of freeze-dried foods during drying is necessary to provide accurate data for prediction of drying rates and times. Although this technique is applied only to beef in this work, it has been successfully used on other foods and should be easily extended to include many products applicable to freeze-drying.

NOMENCLATURE

English Letters		Typical Units
A	area	ft^2
C	constant defined by equation 5.2	$\text{ft g}^2/\text{Btu}$
c_p	specific heat	$\text{Btu}/\text{lb}_m \text{ } ^\circ\text{F}$
\tilde{c}_p	molar specific heat	$\text{Btu}/\text{mole } ^\circ\text{F}$
D_e	effective diffusion coefficient	ft^2/sec
Δh	heat of sublimation	Btu/lb_m
$\tilde{\Delta H}$	molar heat of sublimation	Btu/mole
k	thermal conductivity	$\text{Btu}/\text{hr ft } ^\circ\text{F}$
L	length of dried layer	ft
m_i	initial moisture content	$\frac{\text{grams moisture}}{\text{gram dry meat}}$
m_f	final moisture content	$\frac{\text{grams moisture}}{\text{gram dry meat}}$
N_w	water-vapor molar flow rate	$\text{moles}/\text{ft}^2 \text{ sec}$
P_{w0}	water-vapor partial pressure at surface of sample	lb_f/ft^2
p_0	total pressure at surface of sample	lb_f/ft^2
p_{wX}	water-vapor partial pressure at interface	lb_f/ft^2
p_X	total pressure at interface	lb_f/ft^2
Δp	pressure difference across dry layer	lb_f/ft^2
Q_T	total heat reaching sublimation interface	Btu/hr

English Letters		Typical Units
Q	thermal energy required to increase temperature of vapor stream	Btu/hr
q	heat flux	Btu/hr ft ²
R	total flow rate	grams/hr
r	flow rate from one side of test sample	grams/hr
T	temperature	°F
T _I	inside surface temperature of test sample for special test (see Figure 2)	°F
T _i	interface temperature	°F
T _s	surface temperature	°F
T _X	interface temperature (for Appendix B)	°F
T ₀	surface temperature (for Appendix B)	°F
t	time	hrs
W	accumulated weight loss of dry layer	grams
x	position coordinate in dry layer	ft
X	interface position	ft
y _{w0}	molar concentration of water-vapor at surface of sample	$\frac{\text{moles of water}}{\text{mole of mixture}}$
y _{wX}	molar concentration of water-vapor at interface	$\frac{\text{moles of water}}{\text{mole of mixture}}$
Greek Letters		
\tilde{e}	molar permeability	ft mole/lb _f sec
θ	non-dimensional temperature	dimensionless
ρ	density of water-vapor	grams/cm ³
ρ_i	ice-density	grams/cm ³

Greek
Letters

ρ_s density of solid material
 σ porosity

Typical Units

grams/cm³

dimensionless

Subscripts

1 dry layer in Figures 1 and 2
2 dry layer in Figures 1 and 2

CHAPTER I

INTRODUCTION

Statement of Intent

The need for accurate transport property data on freeze-dried foods is essential to the understanding of heat and mass transfer mechanisms involved in the freeze-drying process. The object of this work was to determine the thermal conductivity of freeze-dried beef during the freeze-drying process. A quasi-steady state analysis was used to determine the thermal conductivity from experimental measurements of the rate of drying and the temperature profile across the dry layer of the product.

The experimental method used is basically that of several previous investigators (5,6,7) but with improvements designed to increase the accuracy and reliability of this method. Also a small improvement has been made in the theoretical analysis by the addition of the convective contribution to heat transfer from the vapor movement through the dry layer.

Freeze-Drying

Freeze-drying is a relatively new process, having evolved in the early 1940's out of the need for the preservation of biological and medicinal substances for which drying time and cost were of little importance. By the late fifties, theoretical and analytical investigations into the mechanism of freeze-drying were begun, with most notable

achievements by Harper and Tappel (1). By 1960, there were at most two major food processors actively involved in marketing freeze-dried foods. In 1964, there were 20 such processors, and the industry is still in its infant stage.

The freeze-drying process has significant advantages over other conventional dehydration processes in the preservation of biological properties, appearance, color and the small shrinkage involved. The high solubility of the product allows quick rehydration whereupon the original color and synthesis are regained. Finally, after cooking, the products are difficult to distinguish from their freshly cooked counterparts. However, freeze-drying is a very time-consuming and costly process; for instance, a 1/2-inch thick sample of meat requires 12 hours to dry to completion. The time involved is costly and represents the primary drawback to this form of dehydration. The porous nature of the dried product makes it an excellent thermal insulator, thus imposing a severe limitation on the rate of heat transfer into the product since the surface temperature is necessarily limited to prevent scorching. If this limitation were removed, drying time could be cut to 10 per cent of the present values according to Harper, et al. (2).

The basic process consists of freezing the product, subjecting it to vacuum conditions of approximately 1 torr (1 mm of Hg) and then supplying heat to the surface of the product whereupon the frozen juices sublime directly to the vapor state due to the low pressure. As heat continues to be conducted to the frozen portion, the ice front recedes into the product. The term "interface" will hereafter be used to refer to the ice front where dried and frozen portions meet. The heat of

sublimation then is conducted through the dried shell to the interface. As heat is conducted to the interface, vapor flows in the opposite direction under the influence of a total and/or a partial pressure gradient. The vapor transport moves under the influence of two driving forces which result in hydrodynamic and diffusional flow. According to Harper, et al. (2), if the total chamber pressure is low compared with the equilibrium vapor pressure of the ice at the ice front, then the primary contribution to vapor movement is hydrodynamic flow. If, however, the total chamber pressure approaches the vapor pressure, the flow reverts to diffusion due to the partial pressure gradient of the water-vapor. Dyer and Sunderland (3) show that the ratio of diffusional to hydrodynamic transport increases as the chamber concentration (moles of water/mole of mixture) decreases, while increasing the total pressure difference has the opposite effect. According to Dyer, then, the total chamber pressure has little effect on the mode of transport but the partial pressure is the determining factor. It is important that the chamber pressure never rise above the triple point of water (4.6 mm Hg) or else the product will begin to thaw. In the case of beef, it is advantageous to have the fiber direction of the product oriented parallel to the heat and vapor transport as this allows the fastest drying time. This is due to the directional dependence of the thermal conductivity of dried beef where the maximum conductivity occurs parallel to the grain. Also, the path parallel to the grain offers the least resistance to the vapor flow.

A thorough understanding of the freeze-drying mechanism requires an accurate knowledge of three important transport properties of the vapor in a porous medium. These are the permeability to flow of vapor

under a total pressure gradient, the diffusivity of water-vapor through air in the porous medium and the thermal conductivity of the porous medium with the water-vapor moving through it. The work of this thesis has been directed toward determination of the thermal conductivity of freeze-dried beef.

Measurement of Thermal Conductivity

General

There are basically two different methods for the determination of thermal conductivity of freeze-dried products. One method involves the determination of conductivity of previously dried samples. This method is a steady-state procedure which is similar to techniques which use the guarded-hot plate. The other method involves the measurement of thermal conductivity during actual freeze-drying processes. Since the freeze-drying process is very slow, a quasi-steady state analysis, which assumes steady conditions over short time intervals, is used to balance the heat conducted through the dried layer against the rate of vapor removal. This method appears to have an advantage over the first one since the conductivity is measured during the actual process and the actual gas mixture is present in the pores of the dried meat. Furthermore, the quasi-steady state method allows much more statistical data to be obtained in a shorter time.

Steady-State Method

The steady-state method has been used by Harper and Sahrigi (4) for measuring the thermal conductivity of freeze-dried beef. The procedure involves using samples of dried beef from 1/2-inch thick to

3/4-inch thick and 2 inches square placed between two lucite plates of known thermal conductivity. These serve as standard references for measuring the heat flow. On the outside of the lucite plates, two circulating water streams are supplied and maintained at approximately 110° and 80°F respectively. With thermocouples placed in appropriate places to measure temperature differences and with measured thicknesses and the known conductivity of the lucite, the conductivity of the sample may be calculated under steady-state conditions. Thermal conductivities were measured by Harper in atmospheres of hydrogen, helium, neon, nitrogen, carbon dioxide, and Freon-12 with pressures ranging from 0.005 torr to atmospheric. For all the gases used, the value of the thermal conductivity is approximately constant up to a pressure of about 0.1 torr. Then the conductivity starts increasing until about 20 torr where it becomes constant again up to atmospheric pressure. According to Harper, et al. (4) "the difference between the high and low pressure values was equal to the pressure-independent conductivity for N₂, CO₂, and F-12, and became less than the pure gas conductivity for the lighter gases."

Quasi-Steady State Method

This method has been used by several investigators including Rolfe (5), Lusk, et al. (6), and Meffert (7). The procedure consists of recording temperatures on the meat sample at the surface and in the center and measuring the weight loss of the sample during the drying process. The basic formulation used by these investigators has been:

$$\frac{kA}{L} (T_s - T_i) = R\Delta h \quad (1.1)$$

where the left side of the equation is the rate of heat conducted

through the dried layer in a short time interval and the right side is the rate of vapor removal due to the sublimation of the ice during the same time interval. The length of the dried layer is related to the cumulative weight loss. The basic assumption made in this type of analysis is that for a short period of time the movement of the interface, the plane where dry and frozen portions meet, is slow enough that the rate of heat conducted to the interface and the rate of sublimation are constant. With the above measurements, the conductivity of the dried layer may be easily determined.

However, the heat absorbed in the sublimation process is not the only way in which heat is absorbed during drying. Other ways in which heat is absorbed are: (1) sensible heat gain of the vapor as it is transported to the surface of the meat; (2) storage of heat in the dried layer; (3) storage of heat in the frozen layer. The quasi-steady analysis ignores the storage of heat in the dried layer. However, this amount is negligible when compared with the heat required for sublimation. When equal drying takes place from both sides of the product, no heat is conducted through the frozen portion, with the result that heat storage in this portion is negligible. However, the heat absorbed by the vapor is significant and can be measured. According to Schneider (8), the temperature profile of a gas in a porous system is very close to that of the solid structure. The heat gained by the vapor between the interface and the surface of the product is given by,

$$Q = R \int_{T_i}^{T_s} c_p dT \quad (1.2)$$

If the specific heat, c_p , is assumed constant then the portion of heat convected toward the surface of the product by the moving vapor may be easily calculated.

Heat Transfer to the Free Surface

For conductivity measurements it is important that a uniform surface temperature be maintained to insure uniform heat conduction through the sample. Previous investigators (5,6,7) using the quasi-steady method in some form have allowed the surfaces of the test samples to be in contact with a tray or some type of support. This means that part of the heat supplied to the test surface is by radiation and part by conduction through direct contact. These methods tend to create some variation in surface temperature on the product due to imperfect contact with trays, heaters or supports. Heat supplied directly to the free surfaces from radiant heaters should help alleviate this problem and is the method used in this work.

Secondary Drying

According to Meffert (7), when the ice phase has disappeared there is still bound or absorbed water that must be removed to give good storage properties to the food. The removal of this moisture is called secondary drying and may involve upwards of 30 to 40 per cent of the original moisture content. Meffert feels that the classical picture of an ice filled layer and an effectively dried layer is erroneous and can lead to serious errors. Specifically, if the calculated thermal conductivity is based only on the ice temperature and does not include the effects of the secondary drying, then the conductivity will be several times higher than that of the bone dry material. The higher apparent

value of the thermal conductivity then should be used with the energy equations based on the two phase model. However, measurements of moisture content on the dried layer of beef during the drying process made by Hatcher (9) indicate that little or no moisture change takes place in this layer after the interface has passed. This would tend to support the classical picture of the distinct dry and frozen layers. The two-phase region is the model used in this thesis.

Thermal Conductivities

Lusk, Karel, and Goldblith (6) report some thermal conductivities for freeze-dried fish obtained during freeze-drying. These are: (1) for salmon, 0.024 Btu/hr ft °F, (2) for haddock, 0.011 Btu/hr ft °F, and (3) for ocean perch, 0.013 Btu/hr ft °F. All of these results were obtained at low chamber pressures (less than 0.1 torr) and are consistent except for salmon, whose higher conductivity is attributed to the use of thick samples which resulted in higher end drying and the resultant departure from an assumption of one-dimensional heat flux.

Rolfe (5) reports a value of 0.033 Btu/hr ft °F for the thermal conductivity of freeze-dried beef obtained under quasi-steady conditions at a chamber pressure of 0.17 torr. For cooked freeze-dried beef, he obtained a value of 0.0376 Btu/hr ft °F.

Meffert (7) reports some conductivities obtained during freeze-drying for various vegetables. The data range in value from 0.0226 to 0.066 Btu/hr ft °F with the majority of the results lying between 0.0306 and 0.0486 Btu/hr ft °F.

Harper and Sahrigi (4), as previously mentioned, report a whole

series of measured thermal conductivities of freeze-dried beef for different gases at varying pressures under steady-state conditions. At total chamber pressures of approximately 0.005 torr the thermal conductivity is about 0.0217 Btu/hr ft °F regardless of the gas in the pores of the meat indicating that heat is conducted through the solid material only. As the chamber pressure rises, the conductivity also rises and becomes dependent on the gas in the pores. Figure 9 on page 46 illustrates the variation of thermal conductivity over a short pressure range for the different gases recorded by Harper, et al (4). It should be noted that at the same chamber pressure the conductivity is higher for the lighter gas. Also since neon has a molecular weight of 20 compared with 18 for water-vapor, it might be expected that these values of thermal conductivity would more closely approach the conductivities during the freeze-drying process. Harper, et al. (4) mention that some measurements were made using water-vapor but no results were given.

In this thesis, values of thermal conductivities for freeze-dried beef are reported for chamber pressures varying from 0.2 to 4 torr. The values are then used in the analytical equations derived by Dyer, et al. (3) to compare theoretical drying times with observed experimental drying times. Also, a test was made to determine the conductivity of previously dried meat by subliming a piece of ice placed between two dry samples. This technique was an attempt to simulate the steady-state method used by Harper, et al. (4) and yet retain the features of the quasi-steady state method.

CHAPTER II

THEORETICAL ANALYSIS

Thermal Conductivity of Freeze-Dried BeefGeneral

After the drying process has begun, the ice layer recedes into the beef thus leaving a dry fibrous layer of meat surrounding the frozen portion. Through this dry, fibrous shell heat must be conducted for the sublimation process. The receding ice front is called the interface and is the plane where the dried and frozen portions meet. This assumption was substantially verified by cutting open partially dried samples for visual inspection. Also, the interface thickness has been measured by Hatcher (9) and found to be less than 5 mm thick. At the interface, the conducted heat provides the necessary heat of sublimation for the frozen layer. The ice phase temperature and the vapor pressure at the interface are dependent on one another and a change in one of these variables results in a consequent change in the other. During freeze-drying, the temperature of the ice phase assumes a value which will result in equilibrium between the heat conducted to the interface and the amount of ice which is sublimated. A change in the temperature or partial pressure of the vapor at the surface of the product or a change in the total chamber pressure will cause the ice phase temperature to change until equilibrium is restored. Furthermore, if the rate of heat transfer is restricted through the dried layer the interface temperature

will become lower, whereas if there is an obstruction to vapor flow then the interface temperature will rise. If either effect is present during a conductivity measurement, then the apparent measured conductivity will be respectively lower or higher than the actual value.

Thermal Conductivity

Heat is conducted simultaneously through the solid portion of the dry shell and through the gas which fills the pores. Kinetic theory shows that the thermal conductivity of an ideal gas is independent of the pressure provided the mean free path of the molecules is much smaller than the dimensions of the confining space. As the pressure is lowered, the mean free path increases. In freeze-drying where low vacuum conditions are present, the mean free path of the vapor molecules have dimensions of the same magnitude as that of the average pore diameters. At a certain pressure, the contribution of the gas towards heat conduction begins to decrease with lowering pressure until heat is conducted solely by the solid. Harper, et al. (4) have shown that the conductivity decreases from a constant value at high pressures to a constant value at low pressures. The difference between the high and low pressure values was equal to the pressure-independent conductivity for the heavier gases.

To determine the thermal conductivity of the dried layer during freeze-drying, an energy balance is made on the dried layer. The heat conducted across the surface of the sample must supply the heat of sublimation for the frozen meat juices, increase the temperature of the vapor as it moves towards the surface and raise the temperature of the dried layer as the interface recedes. However, using the quasi-steady state analysis, described in Chapter I, the energy used in raising the

temperature of the dried layer is neglected. Considering the very light mass of the dried meat this proves to be a valid assumption. According to Dyer (10) the heat storage in the dried layer represents less than one per cent of the total energy input. Therefore with a quasi-steady energy balance the heat conducted through the dry layer minus the heat convected away by the movement of the vapor to the surface equals the heat which is used for sublimation of the frozen portion. Or stated in equation form:

$$\left\{ \begin{array}{l} \text{The rate of heat} \\ \text{conducted through} \\ \text{the dry layer} \end{array} \right\} - \left\{ \begin{array}{l} \text{The rate of} \\ \text{sensible heat} \\ \text{gain by vapor} \end{array} \right\} = \left\{ \begin{array}{l} \text{The rate} \\ \text{of vapor} \\ \text{removal} \end{array} \right\}$$

The assumption of one-dimensional heat and mass flow will subsequently be made and quasi-steady conditions will be assumed to exist. This simply means that the heat and mass fluxes are in one direction only and that over short intervals of time heat and mass fluxes are assumed steady. Also the stored energies of the dried and frozen layers are assumed negligible compared with the heat of sublimation required for the frozen beef juices and the sensible heat gain of the vapor. Figure 1 shows the model used to describe the actual samples under consideration. Flat plate radiant heaters supply the heat for sublimation to the surface of the meat. The entire system is arranged in a vertical orientation to minimize any inequalities of natural convection effects on the two surfaces of the meat.

Quasi-Steady Energy Balance for Freeze-Drying Process

Energy Equation

Heat transfer through the dried layers is assumed to obey

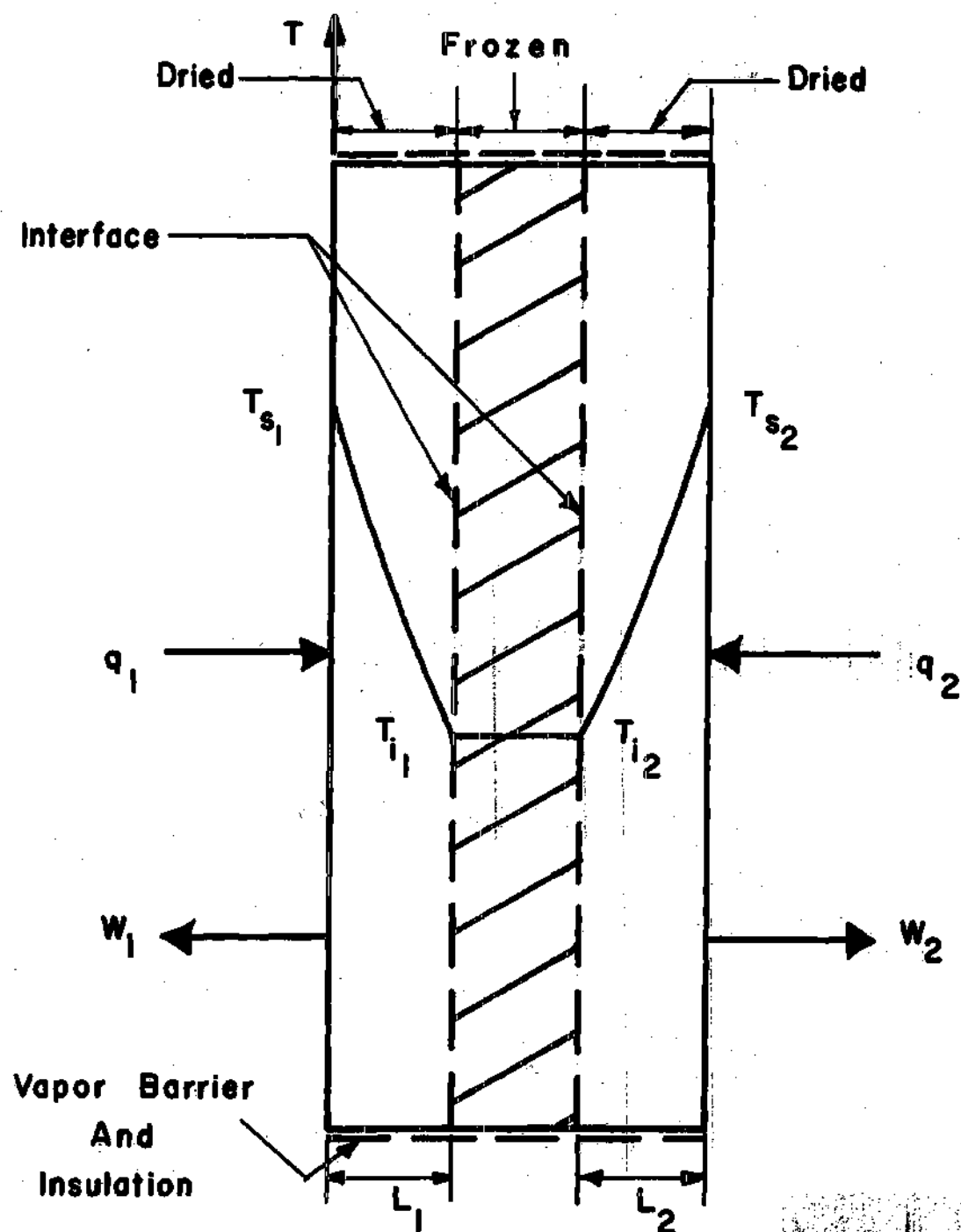


Figure 1. Schematic of Freeze-Drying Model for Thermal Conductivity Analysis.

Fourier's heat conduction equation in one-dimension. Heat is conducted across the dried layer due to the temperature difference between the surface of the product and the interface. At the same time the vapor flowing to the surface through the pores requires heat to raise its temperature. Therefore, neglecting the stored energy in the dried layer, the total heat which reaches the interfaces on both sides is,

$$Q_T = \left\{ k_1 A_1 \frac{(T_{s1} - T_{i1})}{L_1} - r_1 \int_{T_{i1}}^{T_{s1}} c_p dT \right\} + \left\{ k_2 A_2 \frac{(T_{s2} - T_{i2})}{L_2} - r_2 \int_{T_{i2}}^{T_{s2}} c_p dT \right\} \quad (2.1)$$

where the subscript notation refers to Figure 1. The temperature distribution through the dried layer is not linear as indicated in the second and fourth terms of equation 2.1 due to the vapor flux. However, the vapor flow rate is small enough so that the assumption of a linear temperature distribution is valid. Equation B.6 in Appendix B describes the actual temperature distribution in the dried layer. It is shown that the true temperature distribution reduces to an approximately linear distribution for small flow rates. Assuming symmetry, equation 2.1 may be rearranged to give

$$Q_T = 2kA \frac{(T_s - T_i)}{L} - R \int_{T_i}^{T_s} c_p dT \quad (2.2)$$

The first term in equation 2.2 represents the total heat to reach the interface, the second term is the heat conducted across the dry layer

due to the temperature difference and the third term is the heat convected away by the vapor.

The shrinkage of the meat during the drying process is noticeable and should be taken into account in the heat transfer calculations. Therefore the area, A , in equation 2.2 is assumed to be the average area, normal to the heat flow, of the original area of the frozen meat and the final area at the surface (see Figure 1). Since heating takes place at an equal rate from both surfaces of the sample, and since the transient rates of heat transfer in the frozen region are negligible compared with the other energy transfers, the temperature at the center of the sample is assumed to be nearly equal to the interface temperature. This assumption is necessary because of the relative ease of measuring the center or frozen portion temperature as compared with measuring the actual interface temperature. Henceforth, the center temperature will be referred to as the interface temperature.

The energy conducted to the interface then supplies the heat for sublimation of the frozen layer. This energy is represented by

$$Q_T = R \Delta h \quad (2.3)$$

Combining equations 2.2 and 2.3 gives

$$R \Delta h = 2kA \frac{(T_s - T_i)}{L} - R \int_{T_i}^{T_s} c_p dT \quad (2.4)$$

The thickness of the dried layer, L , is determined in the following manner. The total moisture loss of the dry layers on both sides of the sample is given by the following relationship:

$$W = \rho_s (m_i - m_f) (A_1 L_1 + A_2 L_2) \quad (2.5)$$

Due to symmetry, equation 2.5 may be rearranged to give

$$W = 2AL\rho_s (m_i - m_f) \quad (2.6)$$

where, A , is again taken as the average area normal to heat and vapor flow. Therefore, the product, AL , represents the average volume of the dried layer, ρ_s is the average density of the dried layer and m_i and m_f are the initial moisture content of the meat per unit mass of solid material and the final moisture content respectively. Substitution of equation 2.6 for L in equation 2.4 and rearrangement gives the following equation which is used for the determination of the thermal conductivity of the dried layer.

$$R = \frac{4A^2 \rho_s (m_i - m_f)}{\Delta h + \int_{T_i}^{T_s} c_p dT} k \frac{(T_s - T_i)}{W} \quad (2.7)$$

Property Measurement and Calculation

The density of the solid material, ρ_s , is determined by drying the sample to completion and then weighing it and carefully measuring its final dimensions. With this information and the initial dimensions of the sample, the density of the solid material before and after drying may be calculated. The average of these two values, ρ_s , is used in equation 2.7. The initial moisture content, m_i , is also determined at the completion of the drying cycle from the moisture loss and the weight of the bone dry meat. The final moisture content is assumed to

be zero. The surface and center temperatures are measured with small gage copper-constantan thermocouples. The accumulated weight loss is measured gravimetrically during the drying process with a Mettler balance.

Nearly all investigators in the field of freeze-drying research have assumed that the frozen juices of a food product have nearly the same physical properties as pure ice. For this reason thermodynamic and heat transfer calculations involving the sublimation of the frozen juices to the vapor state have employed the heat of sublimation for pure ice which is 1220 Btu/lb_m. Dyer, et al. (11) have recently made a series of measurements of the equilibrium vapor pressure of the frozen liquid in meat. They found that the equilibrium vapor pressure is depressed below that of pure ice at the same temperature. One consequence of this result is that from the Clausius-Clapeyron equation,

$$\frac{d \ln p}{d \frac{1}{T}} = -\Delta h/R \quad (2.8)$$

it is found that the heat of sublimation for the frozen juices is 1488 Btu/lb_m which is approximately 22 per cent higher than pure ice. The experimental results of this work presented in Chapter V employ both heats of sublimation mentioned above for comparison sake.

The gas present in the pores of the dry layer of beef during freeze-drying is a binary mixture of air and beef juice-vapors. At the interface, the movement of the air is zero and hence there is essentially no movement of the air throughout the dry layer. The vapor however flows from the interface to the surface and its temperature

increases at the expense of the thermal energy conducted from the surface to the interface. Hence, the specific heat employed in equation 2.7 should be that of the vapor component only. The value used for this work is that of water-vapor which is $0.44 \text{ Btu/lb}_m \text{ } ^\circ\text{F}$ since specific heat data for the actual vapor is lacking.

To determine the conductivity, k , during freeze-drying, the flow rate, R , and the accumulated weight loss, W , are measured over short time intervals (30 minutes). A plot of R versus $(T_s - T_i)/W$ is then made, and as can be seen from equation 2.7 the slope of this curve will give the thermal conductivity.

The analysis made in this chapter is an idealized one and in the actual case it can be subject to numerous errors. It is necessary that the dimensions of the product be accurately measured as with the small samples used any error can be quite significant. Properties such as the density and moisture content must be accurately measured. For this work, the value of each used in the final calculations was the average of many samples. This procedure was deemed the most reliable as any individual measurement might be subject to a wide error. The temperature measurements were tedious to make and again were subject to serious error. Finally the assumption of one-dimensional heat transfer and parallel vapor flow were the most exacting requirements to reproduce experimentally and the most important conditions to maintain. The experimental results based on the analysis of this chapter are presented in Chapter V.

Alternate Method for Conductivity Measurement

The results of Chapter V indicate thermal conductivities approximately 30 per cent higher than those measured by Harper, et al. (4) for

neon. As was mentioned in Chapter I, there is reason to believe that the values Harper obtained for neon might approach those obtained under freeze-drying conditions. For this reason, it was decided to attempt the measurement of thermal conductivity of a dried sample under conditions that would closely approximate freeze-drying and yet simulate the steady-state method used by Harper. To do this a piece of pure ice was placed between two dry samples of beef (see Figure 2). This arrangement was then put into the freeze-dryer and the ice was allowed to sublimate between the beef samples. Knowing the thickness of the dried samples, the temperature of the surfaces and the rate of sublimation the conductivity of the dried samples was easily calculated.

The method for calculation of the conductivity requires essentially the same analysis used for freeze-drying. The heat transfer through the two dry samples is related to the vapor removal by,

$$k_1 A_1 \frac{(T_{s1} - T_{I1})}{L_1} + k_2 A_2 \frac{(T_{s2} - T_{I2})}{L_2} = R \Delta h + R \int_{T_I}^{T_s} c_p dT \quad (2.9)$$

where if symmetry is assumed,

$$2kA \frac{(T_s - T_I)}{L} = R \Delta h + R \int_{T_I}^{T_s} c_p dT \quad (2.10)$$

The notation is the same as that used previously except, T_I , which is the temperature on the surface of the sample near the ice. Again it is

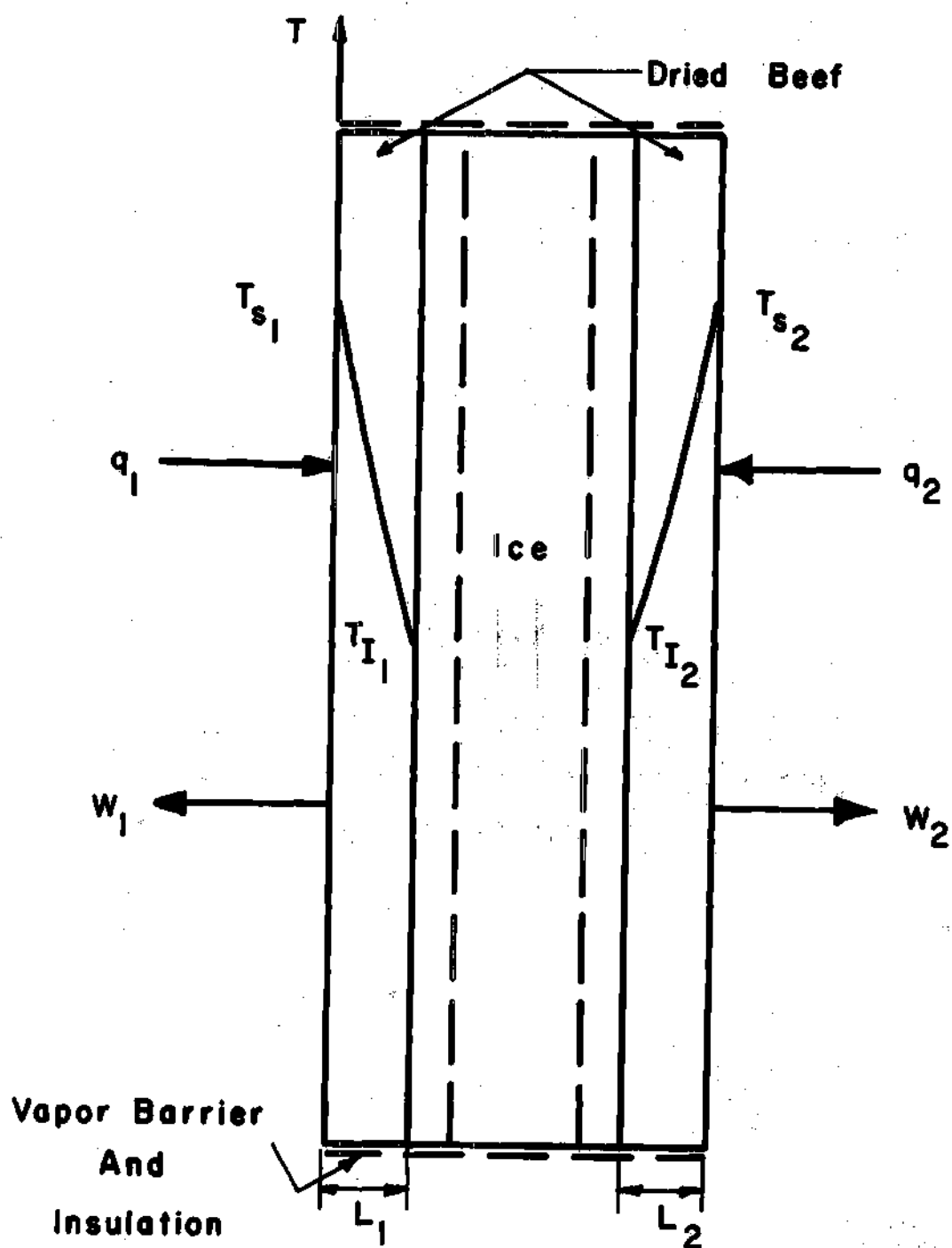


Figure 2. Schematic of Test Sample for Special Thermal Conductivity Test with Ice Between Two Dry Beef Samples.

necessary to obtain data over short time intervals as some decrease in R may occur as the drying proceeds. Results for this method are listed in Chapter V with the data obtained during freeze-drying.

CHAPTER III

EQUIPMENT AND INSTRUMENTATION

Equipment

The basic components of the equipment used in this work are a vacuum chamber with a vacuum pump and a condenser. The equipment layout is shown in Figure 3. The equipment specifications are given in Appendix C.

Vacuum Chamber and Heaters

The vacuum chamber consists of a two foot cubical chamber fabricated from 1/4-inch steel plate and welded about an internal frame of angle iron. The door is made from 1/2-inch steel plate and rides on slotted hinges, which allow horizontal movement from front to back, so that it may be drawn up tight. Two 4 inch square port holes are provided in the door for observation into the chamber. A natural rubber gasket glued around the frame of the entrance provides a vacuum tight seal. The door is drawn up tight using 20 - 3/8 inch steel bolts which pass through the outer edge of the door and a flange welded to the front of the chamber.

Two plate type 110 volt, 1700 watt electric radiant heaters mounted vertically on angle iron brackets provide the heat of sublimation during drying.

Vacuum Pump and Condenser

A rotary type vacuum pump removes non-condensable gases from the chamber by discharging them to the atmosphere thus serving to develop

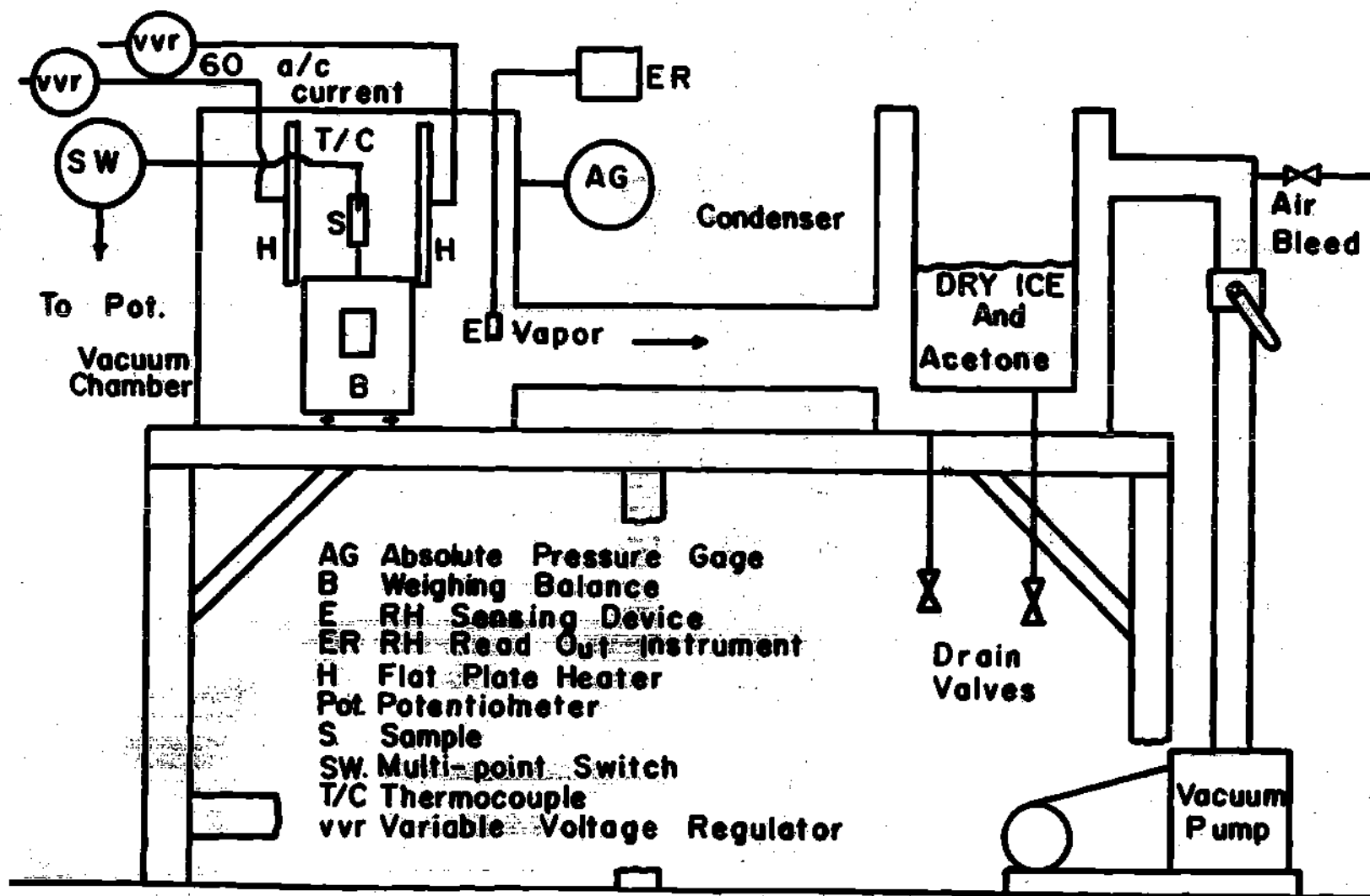


Figure 3. Schematic Diagram of Freeze-Drying Test Equipment.

a vacuum and maintain it.

The condenser serves as a cold trap between the vacuum chamber and the pump. It consists of two concentric cylinders with the inner one open at the top to allow the condenser to be cooled by dry ice and acetone. This provides a cold surface of approximately -100°F for condensing the water-vapor removed from the drying sample. A six inch diameter pipe carries the water-vapor from the vacuum chamber to the outer cylinder of the condenser where the vapor comes in contact with the cold surface and is frozen out. The non-condensable gases pass around the inner cylinder and on to the vacuum pump. The condenser also serves as a secondary pump due to its low temperature which creates a pressure gradient between the chamber and condenser.

The condenser thus serves the dual purpose of reducing the volume of gases handled by the vacuum pump and preventing the water-vapor from entering the pump. Water-vapor which enters mechanical vacuum pumps often contaminates the pump oil and may cause mechanical difficulties. Also, the low chamber pressures required could not be obtained with the vacuum pump used without the condenser.

Instrumentation

Sample

The meat samples used in the thermal conductivity tests were taken from utility round beef purchased from a local packing house. This variety was selected for its very lean texture. The samples were then cut to minimize fatty inclusions and to orient the fibers of the beef parallel to the expected heat and vapor flow. The meat was frozen

and then cut on a band saw into samples 1/2-inch thick and 3 inches in diameter. Samples 1/2-inch thick were used in order to minimize end drying but still allow enough drying time to obtain sufficient data. The diameter of 3 inches was about the maximum that could be cut and still have a uniform sample.

Temperature and Pressure Measurement

Thirty-six gage copper-constantan thermocouples were selected for the measurement of meat surface and center temperatures. The small diameter wire was necessary to prevent large conduction errors. The thermocouple leads inserted into the center of the sample were passed once around the rim of the sample to provide a long isothermal path to further minimize conduction errors. The leads from the meat sample were attached to quick-disconnect thermocouple plugs which allowed fast hookup with the wires leading outside the chamber during startup. The leads were passed through the wall of the chamber using standard vacuum feed-through devices. Outside the chamber a multipoint switch and a Leeds and Northrup potentiometer, Model 8662, provided the necessary readout equipment for the temperatures. A 32°F reference junction was provided using crushed ice and water in a thermos bottle.

Other temperatures were measured at the heater surfaces, insulation surfaces and the humidity sensing element.

The vacuum chamber pressure was measured using a Wallace Tiernan absolute pressure gage with a range of 0.1 to 20 torr in 0.1 torr increments. This type of gage is a direct-reading, diaphragm type, mechanical, absolute pressure gage which is self-compensating for variations in both ambient pressure and temperature. Its range, accuracy and simplicity

make it particularly suitable for this type of work.

Humidity Measurement

Relative humidity in the chamber was measured at the entrance to the pipe leading from the chamber to the condenser. An element sensitive to partial pressure of the water-vapor made by Hydrodynamics, Inc. of Baltimore, Maryland, was used to measure the relative humidity. The output from the element was indicated on an instrument made by the same manufacturer and converted to relative humidity from a factory calibrated curve. Leads from the element were fed through the chamber wall with devices similar to those used for the thermocouples.

Weighing Balance

A precision Mettler laboratory balance was used to measure weight loss of the sample during the drying process. The scale is calibrated from 0 - 1000 grams. A built in tare weight increases the total capacity to 2000 grams. The pan is magnetically dampened and the dial is illuminated so that readings were easily obtained through the port holes of the chamber door.

Estimate of Instrument Errors

The absolute pressure gage used to measure the vacuum chamber pressure was relatively new and its calibration curve, was furnished by the manufacturer. The maximum error in the measurement of the vacuum chamber pressure is estimated to be ± 0.01 torr.

All thermocouples were used with a 32°F reference junction temperature maintained by an ice-water bath. The potentiometer is calibrated in increments of 0.005 millivolts. Thus the maximum error due to the potentiometer, in the various temperature measurements is estimated

to be $\pm 0.25^{\circ}\text{F}$. The total error is estimated to be no greater than $\pm 1.0^{\circ}\text{F}$.

According to Mayne (14) the humidity sensing element is accurate to within 5 per cent of the calibration curves supplied by the manufacturer.

The precision balance is calibrated in one gram increments which could be interpolated easily within 0.1 gram. Also, the balance indicator and scale are in the same plane thus avoiding parallax error. In addition, the balance was checked at the beginning of each test by placing known weights on the balance and adjusting the scale for zero error. Thus the estimated maximum error in weighing is estimated to be ± 0.1 gram.

CHAPTER IV

EXPERIMENTAL PROCEDURE

Introduction

A number of tests were conducted to determine the thermal conductivity of freeze-dried beef. The tests were made at chamber pressures ranging from 0.2 to 4.0 torr to determine the effect of absolute pressure on thermal conductivity. The time required for each test was approximately three to four hours as the thin samples used required only a short time to dry.

Pretest Procedure

The meat used for the tests was utility round beef. It was selected primarily for its lack of fatty inclusions which can significantly affect the freeze-drying process. According to Rolfe (5) lean beef contains approximately 70 to 75 per cent water by weight, whereas fat contains only 8 to 10 per cent water. This difference results in the fatty tissue drying faster because less heat is required, and when dry the fat has a higher thermal conductivity due to the small voids present. This will result in higher temperatures for the fatty inclusions. Rendering or melting of the fat may occur causing it to spread out into the porous structure of the dry lean meat. This will then inhibit the vapor removal from the drying sample and cause the temperature of the frozen layer to increase. Then, as mentioned in Chapter II, the measured conductivity will be higher than that of the lean meat.

The fresh meat was cut with a knife to approximately its final shape. This step was critical in that the meat had to be cut so that when frozen the fiber orientation would be uniform and parallel to the heat and vapor fluxes. After the sample had frozen, it was cut to its final shape of 3 inches in diameter and 0.5 inches in thickness on a band saw. To prevent serious thawing, the sample was placed on a wood base and was secured with a wood clamp during this cutting procedure.

After the sample had been cut to final form, a small hole was drilled radially from the edge to the center of the sample for a thermocouple. The sample was then weighed to determine its initial weight. Then a thermocouple was inserted into the center of the sample and the leads were passed once around the rim of the sample to provide as long an isothermal path as possible to prevent conduction errors in the thermocouples. The edge was wrapped with plastic wrap and tape to provide a vapor seal. To retard end drying as much as possible, fiberglass insulation was placed around the edge of the sample. The insulation was cut from a rigid block of insulation 1.5 inches thick, circular in shape and with an overall diameter of 7 inches. A concentric hole 3 inches in diameter was cut from the center to accommodate the sample. Then it was cut on a diameter into two sections so that it could be placed around the meat sample. Masking tape wrapped around the rim of the insulation served to hold the two pieces together. Finally, thermocouples were imbedded immediately under the exposed surfaces of the sample to measure the surface temperatures. Surface temperatures are difficult to measure accurately. Small gage thermocouple wire (36 gage) was used to minimize the conduction error along the thermocouples. To prevent radiant heat

from impinging directly on the wires, the leads were shielded from the heaters by passing them first through the insulation and then embedding them just under the meat surface up to and including the junction.

Part of the procedure required that the sample be positioned in a vertical manner in the vacuum chamber between two vertical heaters. To accomplish this, it was necessary to remove the existing superstructure of the Mettler scales which includes the weighing pan and its support. A lucite dowel pin was constructed so that it could support the sample and its insulation vertically and fit onto the place provided for the regular equipment. The prepared sample is shown diagrammatically in Figure 4.

To further retard radial heat flux to the test sample, two blocks of fiberglass insulation, 1-1/2 inches thick, were cut to the size of the flat plate heaters. In the center of each of these a hole a little larger than the diameter of the meat sample was cut. Then each block of insulation was suspended about a 1/4-inch in front of each heater with aluminum foil on the surfaces near the heaters. Then the meat sample was placed between these blocks so that the meat could "see" the heaters through the holes in the insulation. These large blocks of insulation served to retard radiant heat transfer to the surfaces of the insulation surrounding the rim of the meat sample in turn reducing the radial heat flux to the meat and preventing end drying of the test sample.

Test Procedure

The following procedure was used during the test runs for determination of the thermal conductivity of freeze-dried beef.

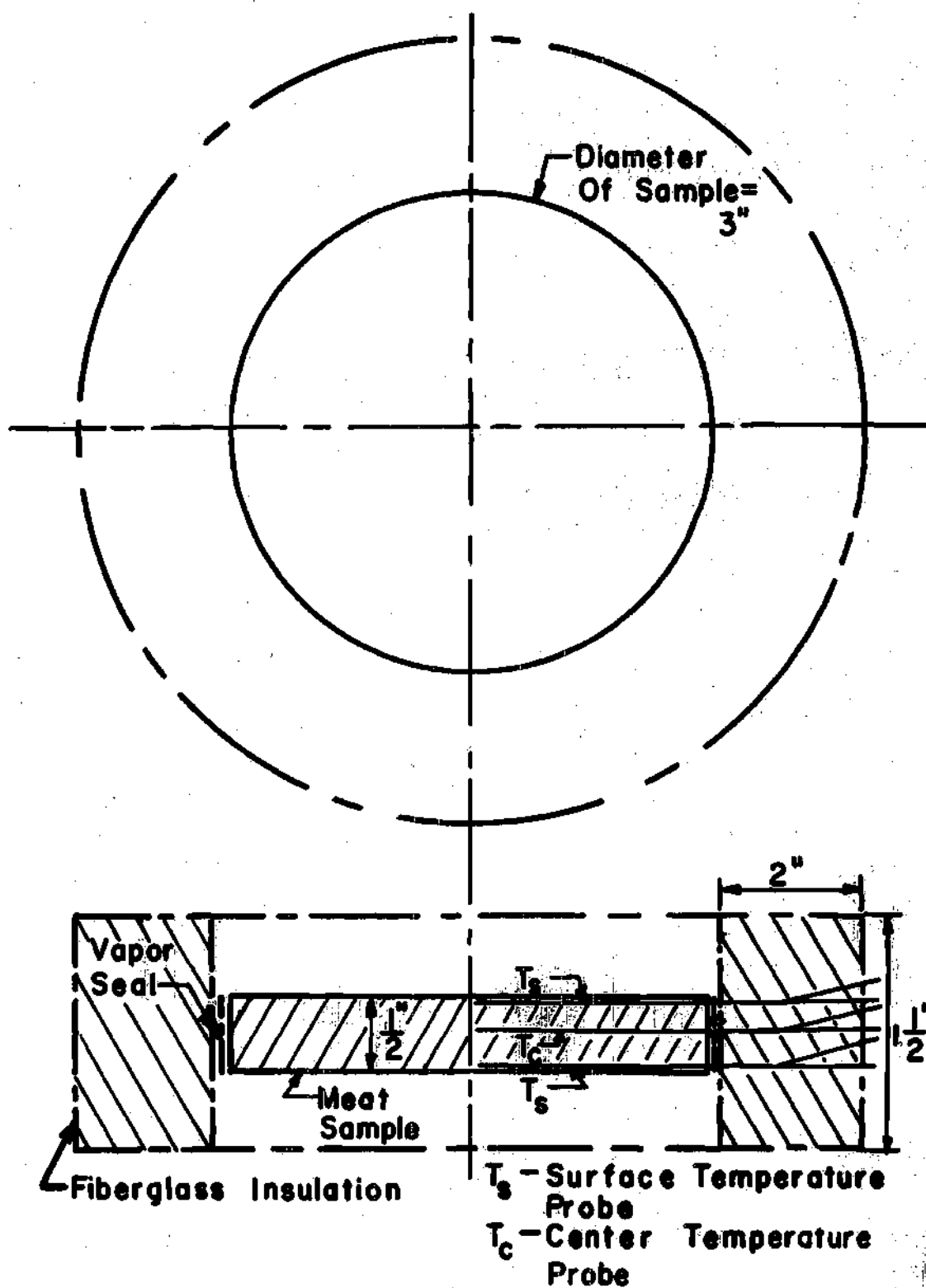


Figure 4. Schematic of Prepared Sample for Thermal Conductivity Test.

1. Dry ice and acetone were placed in the condenser to provide a cold surface for condensing water-vapor and to help develop and maintain the vacuum in the drying chamber.
2. The sample with its fiberglass holder was placed on the scales and centered between the heaters.
3. The fiberglass insulation blocks were suspended in front of the heaters in pre-designated positions.
4. The humidity sensing element was connected to its leads.
5. All thermocouple connections were made and checked for proper working order.
6. The door to the chamber was closed and bolted shut.
7. The initial weight of sample, including insulation and thermocouple wires was recorded.
8. The vacuum pump was turned on and the chamber was evacuated to the desired pressure.
9. Precise control of the chamber pressure was obtained by bleeding air into the system through a needle valve located between the condenser and the pump.
10. The heaters were turned up to the maximum permissible power to raise the meat surface temperatures as fast as possible.
11. Initial temperature readings were recorded as well as the starting time.
12. The surface temperature's of the meat were continuously checked during the warm-up period and the powerstats supplying current to the heaters were adjusted to keep the temperatures approximately equal.
13. As the surface temperatures approached 100°F which usually

took 30 to 45 minutes, the powerstats were turned down low to prevent overheating of the surfaces.

14. When the surface temperatures reached 100°F the temperatures, weight and relative humidity were recorded at 15 minute intervals until the end of the test.

15. The powerstats for the heaters were periodically adjusted to maintain the meat surfaces at 100°F.

16. The air bleed valve required occasional adjustment to maintain the chamber pressure at the desired level.

17. Dry ice was periodically added to the condenser to maintain it at a low temperature.

Post Test Procedure

For some of the test runs the sample was removed from the dryer before it had completely dried. This was done to observe the uniformity with which the sample dried, that is, how closely the drying approximated the one-dimensional model.

For complete drying the sample was allowed to remain in the dryer until the temperatures were equal throughout the sample and an hour passed in which no weight loss was recorded. Again the sample was cut open for observation to determine if any non-uniformities were present. In both cases, the fiber orientation was observed to insure that it was parallel to the heat and vapor flow. Also, the final weight was recorded as a check against the weight loss observed during drying.

Test Procedure for Alternate Method of Conductivity Measurement

This test required two thin pieces of previously dried beef and a

piece of pure ice placed between them. Two frozen beef samples were first prepared by cutting them to a 3 inch diameter and 1/4-inch thickness. These were dried completely in the vacuum chamber and then carefully measured for the heat transfer calculations. Next, a piece of ice of nearly the same dimensions as the meat samples was prepared by freezing distilled water. Thermocouples were placed on each side of the two beef samples. Then very quickly the piece of ice with the dry beef samples on either side were taped together. Insulation was placed around the sample and the whole arrangement was placed in a freezer until the test was ready to be run.

The basic test procedure was essentially the same as that followed during the freeze-drying procedure. The test sample was placed vertically in the chamber with the outside surfaces of the beef facing each flat plate heater. Thus, thermal radiation striking the meat surfaces was transferred through the dry beef samples to the ice which then sublimates. The vapor flowed through the beef samples countercurrent to the heat flow until it reached the chamber. The only difficulty experienced was in controlling the surface temperatures of the sample which were very sensitive to slight changes in the heater power. However, with close attention and care this problem was easily overcome.

After sufficient time had passed to obtain enough data, the sample was removed from the vacuum chamber for observation. The test sample was in good condition and the piece of ice had sublimated very uniformly.

CHAPTER V

PRESENTATION AND DISCUSSION OF RESULTS

Calculation Procedure

The thermal conductivity of freeze-dried beef was measured at the following total chamber pressures.

Chamber Pressure

torr

0.2

0.5

0.7

1.0

2.0

3.0

4.0

The data obtained in each test are plotted in Figures 5 through 7. The flow rate is plotted against the temperature difference across the dry layer divided by the accumulated weight loss. Each datum point was obtained by calculating the average flow rate over a thirty minute period. The accumulated weight loss was that which had occurred at the mid-point of each thirty minute period. The periods were overlapped to obtain as many data points as possible. The surface temperature of the sample was raised to 100°F within 30 to 45 minutes of the start of the test run, and the frozen portion temperature (henceforth the interface

temperature) remained virtually constant throughout each run. Therefore, it is assumed that the temperature difference was constant during the run.

Equation 2.7 of Chapter II, the governing equation for the determination of the conductivity, may be written as,

$$R = Ck \frac{(T_s - T_i)}{W} \quad (5.1)$$

where

$$C = \frac{4A^2 \rho_s (m_i - m_f)}{\Delta h + \int_{T_i}^{T_s} c_p dT} \quad (5.2)$$

The term, C , was assumed to be a constant throughout each run. Each factor in equation 5.2 was determined in the following manner:

1. The cross-sectional area, A , normal to the heat flux was assumed to equal the average of the values for the frozen sample at the beginning of the run and the dried sample at the conclusion of the run. This value was normally about 6.75 in^2 . The total change in area was approximately 0.6 in^2 .

2. Due to the slight shrinkage of the beef samples during the drying process, the density, ρ_s , of the solid material changes continuously. Therefore the density of the solid material was determined both before drying as 0.24 g/cm^3 and after drying as 0.28 g/cm^3 . This was determined through consideration of the dimensions before and after drying and the final dried weight. The average of these two values, 0.26 g/cm^3 , was used in equation 5.2.

3. The initial moisture content, m_i , may be determined from the initial and final weight of the meat sample. The value obtained in this work was on the average 3.17 g moisture/g dry meat.

4. The final moisture content, m_f , of the dried layer was assumed to be zero. There may however be 2 or 3 per cent of the initial moisture content which remains in the part of the dried layer near the sublimation interface.

5. As mentioned in Chapter II, two values are used for the heat of sublimation, Δh . For pure ice, $\Delta h = 1220 \text{ Btu/lb}_m$; for frozen meat juices Dyer, et al. (11) calculated a value of 1488 Btu/lb_m for Δh .

6. The specific heat is assumed to equal the value for water-vapor which is $0.44 \text{ Btu/lb}_m \text{ } ^\circ\text{F}$. With c_p a constant, the integral in equation 5.2 becomes $c_p(T_s - T_i)$.

Experimental Results

The measured values of the thermal conductivity of freeze-dried beef along with other pertinent information are listed in Table 1. Figures 5 through 7 are representative of the curves obtained during the conductivity tests. The tabulated results of each test are listed in Appendix A. In general, these curves exhibit the same characteristics as those noted by Lusk, et al. (6). That is, at the beginning of each run when the flow rates are highest and conditions are far from quasi-steady, the points are higher than the average. During most of the run, the points lie near a straight line passing through the origin as would be expected. Near the end of each test run, the data points begin falling off lower than the average. This may be caused by the increasing ratio

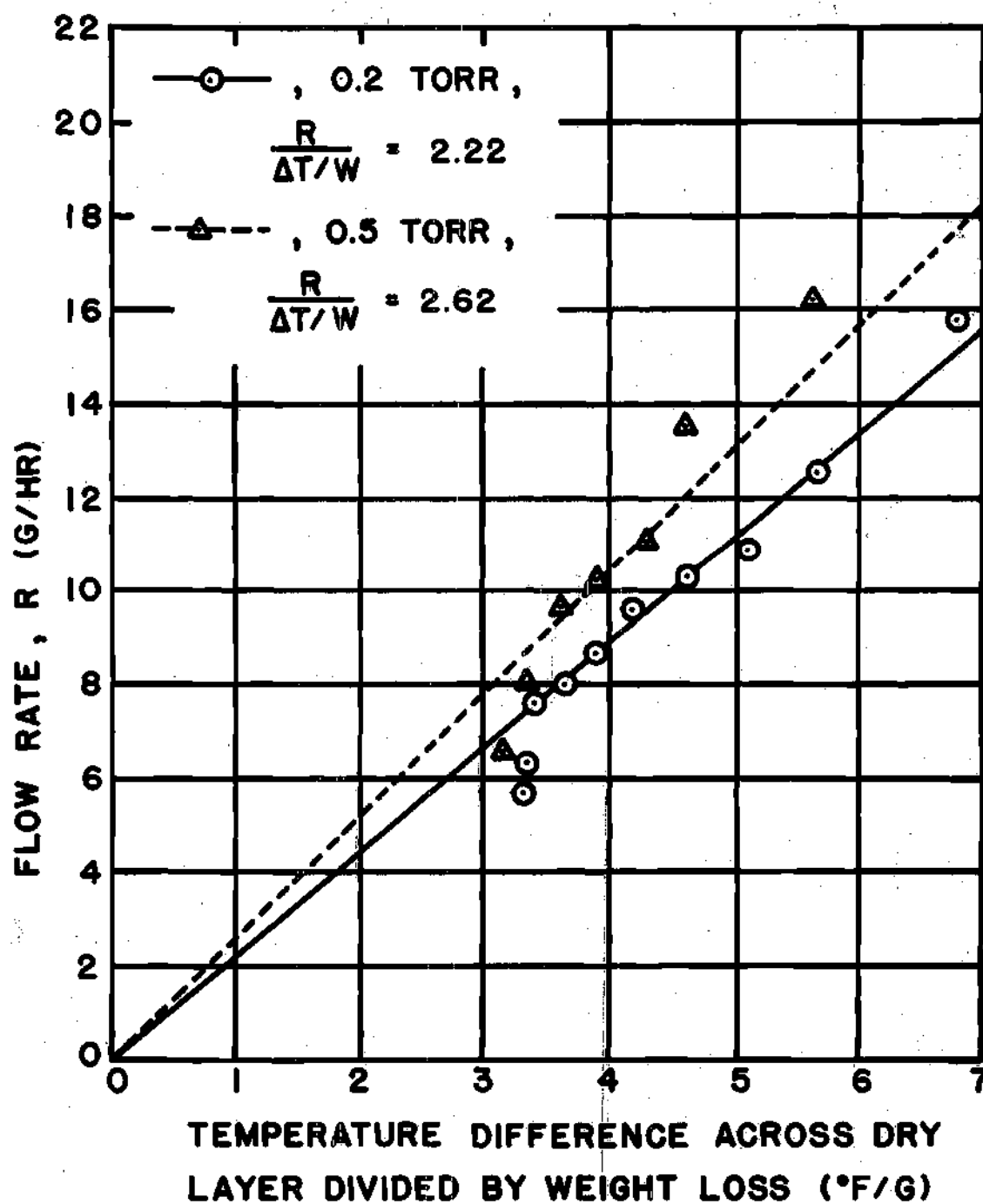


Figure 5. Plot for Determination of Thermal Conductivity, 0.2 torr and 0.5 torr.

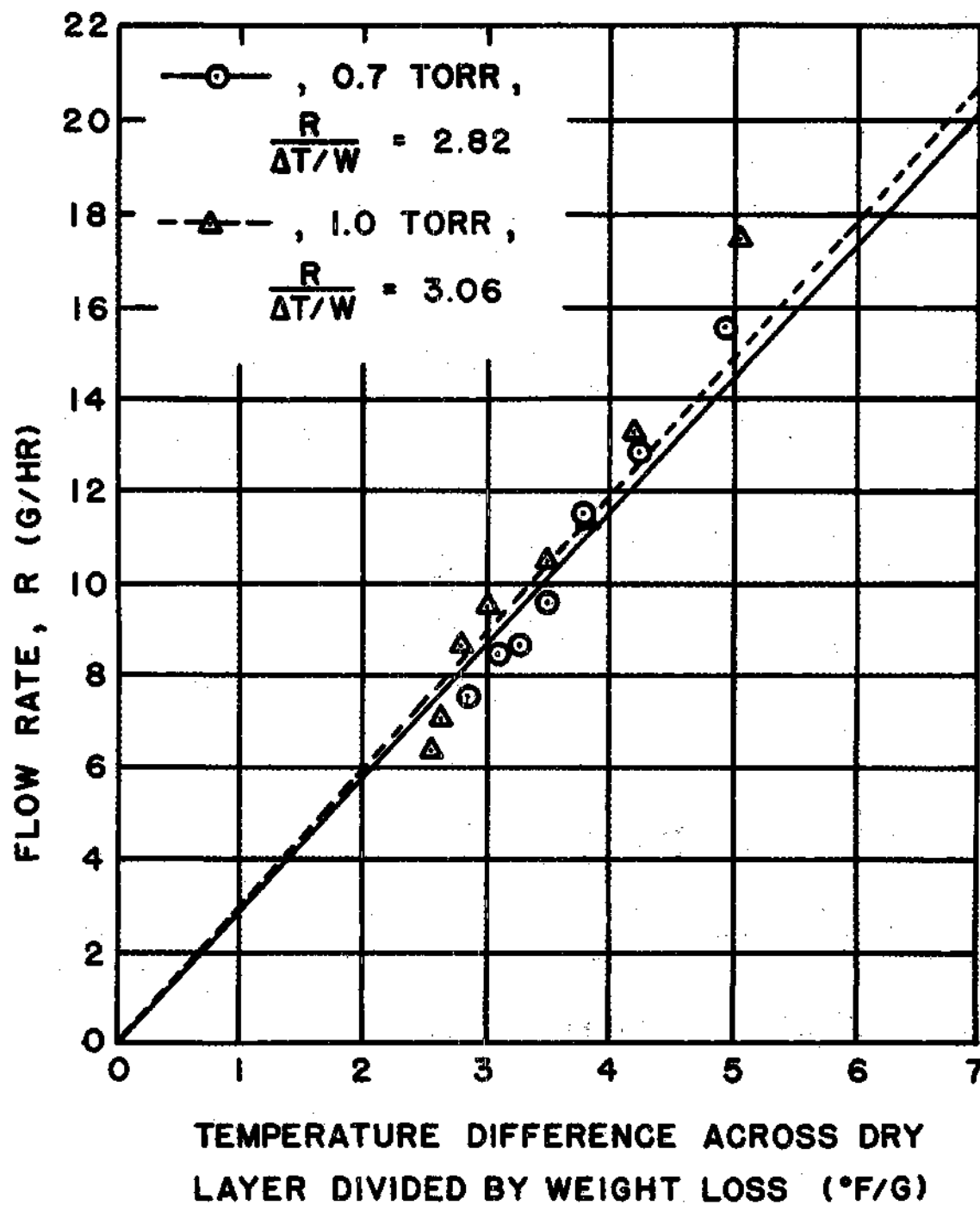


Figure 6. Plot for Determination of Thermal Conductivity, 0.7 torr and 1.0 torr.

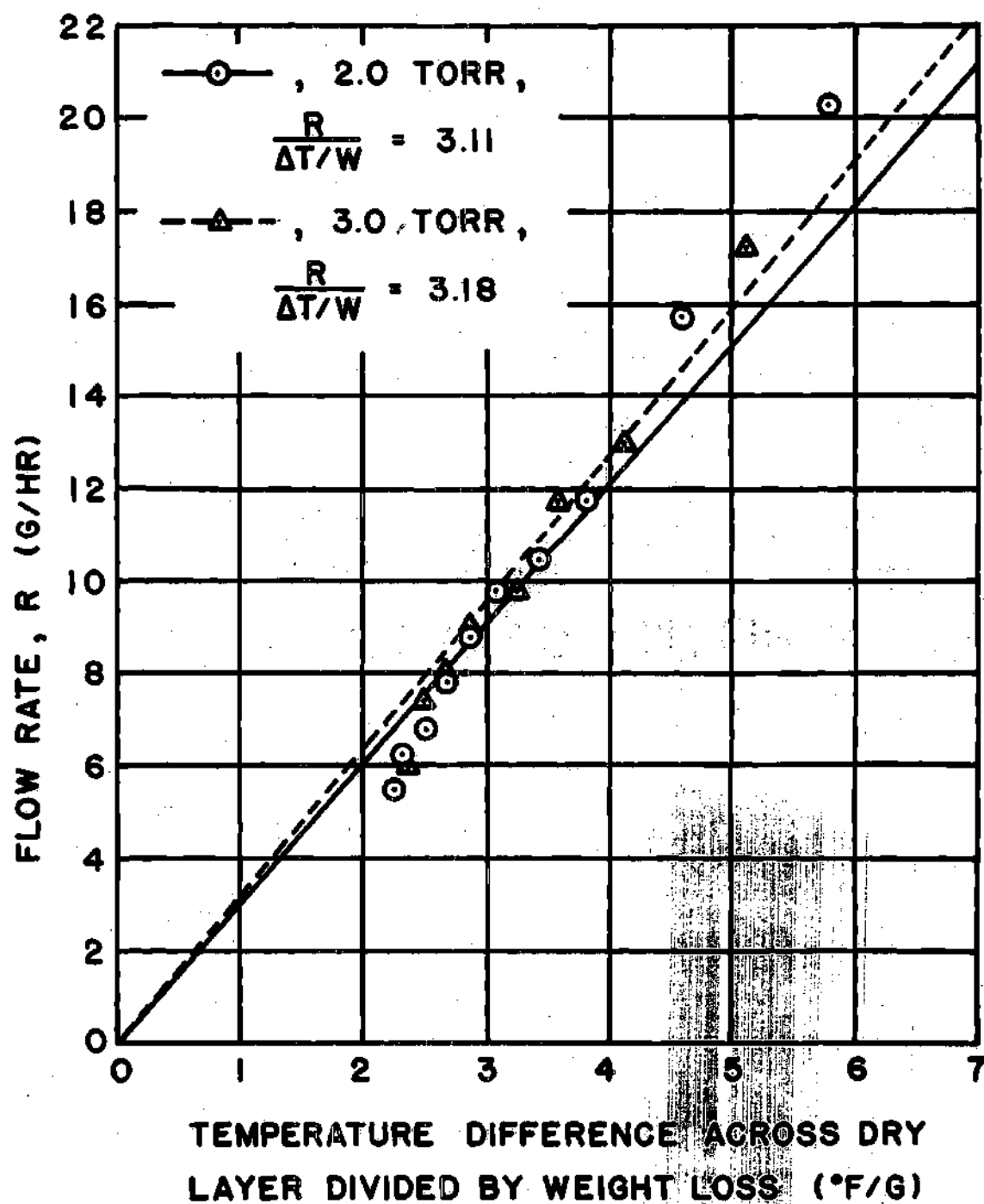


Figure 7. Plot for Determination of Thermal Conductivity, 2.0 torr and 3.0 torr.

of the time rate of change of internal energy of the dried layer to the energy of sublimation. This ratio is small during most of the drying process but becomes larger during the final stages of drying. With decreasing chamber pressure, the slope of the curves and consequently the thermal conductivity decrease since the flow rates are lower and temperature differences across the dried layer are larger. The slope of each curve is shown on the respective graph.

Table 1. Results of Thermal Conductivity Tests on Freeze-Dried Beef

Chamber Pressure torr	$\frac{R}{\Delta T/W}$ $\frac{g^2}{^\circ F/hr}$	C^* $\frac{ft\ g^2}{Btu}$	C^{**} $\frac{ft\ g^2}{Btu}$	k^* $\frac{Btu}{hr\ ft^\circ F}$	k^{**} $\frac{Btu}{hr\ ft^\circ F}$	k^* without convective contribution
0.2	2.22	72.7	60.1	0.0305	0.0369	0.0291
0.5	2.62	73.1	60.5	0.0338	0.0433	0.0344
0.7	2.82	73.4	60.6	0.0384	0.0465	0.0370
1.0	3.06	73.5	60.7	0.0416	0.0503	0.0401
2.0	3.11	73.9	60.9	0.0421	0.0510	0.0408
3.0	3.18	74.1	61.1	0.0429	0.0520	0.0417

where:

C^* and k^* are the constant C (equation 5.2) and k evaluated using $\Delta h = 1220\ Btu/lb_m$.

C^{**} and k^{**} are the constant C and k evaluated using $\Delta h = 1488\ Btu/lb_m$.

Table 1 shows the results of the conductivity tests calculated using both the heat of sublimation of pure ice (1220 Btu/lb_m) and the heat of sublimation of frozen beef juices (1488 Btu/lb_m). The results using the Δh of 1488 Btu/lb_m are about 22 per cent higher than the results which use the heat of sublimation of pure ice. However, as will be seen later, this difference does not materially affect predicted drying times from drying rate equations due to a nearly linear relationship between the conductivity and the heat of sublimation.

Figure 8 compares the conductivity with the chamber pressures and illustrates the relatively smooth increase of the conductivity with increasing chamber pressure. Also, for certain chamber pressures extra tests were run and these results are shown, and as can be seen the agreement between different samples is good. Note that the sharp decrease in conductivity below a chamber pressure of 1 torr is evidently due to the decreased contribution of the vapor toward heat conduction mentioned in Chapter II. Above 1 torr, the pressure dependence of the conductivity is much less pronounced.

Table 2 illustrates the wide range in temperature of the interface of the samples with chamber pressure. At the chamber pressure of 4 torr, the interface temperature was 28.5°F . This combination of total pressure and temperature was either very near or above the critical point of the frozen beef juices allowing at least partial thawing of the product during drying. At the end of the test, the product had undergone considerable shrinkage, was highly pitted and was reddish in appearance. The calculated thermal conductivity was $0.061 \text{ Btu/hr ft}^\circ\text{F}$ (for $\Delta h = 1220 \text{ Btu/lb}_m$) which is considerably higher than expected. The various factors

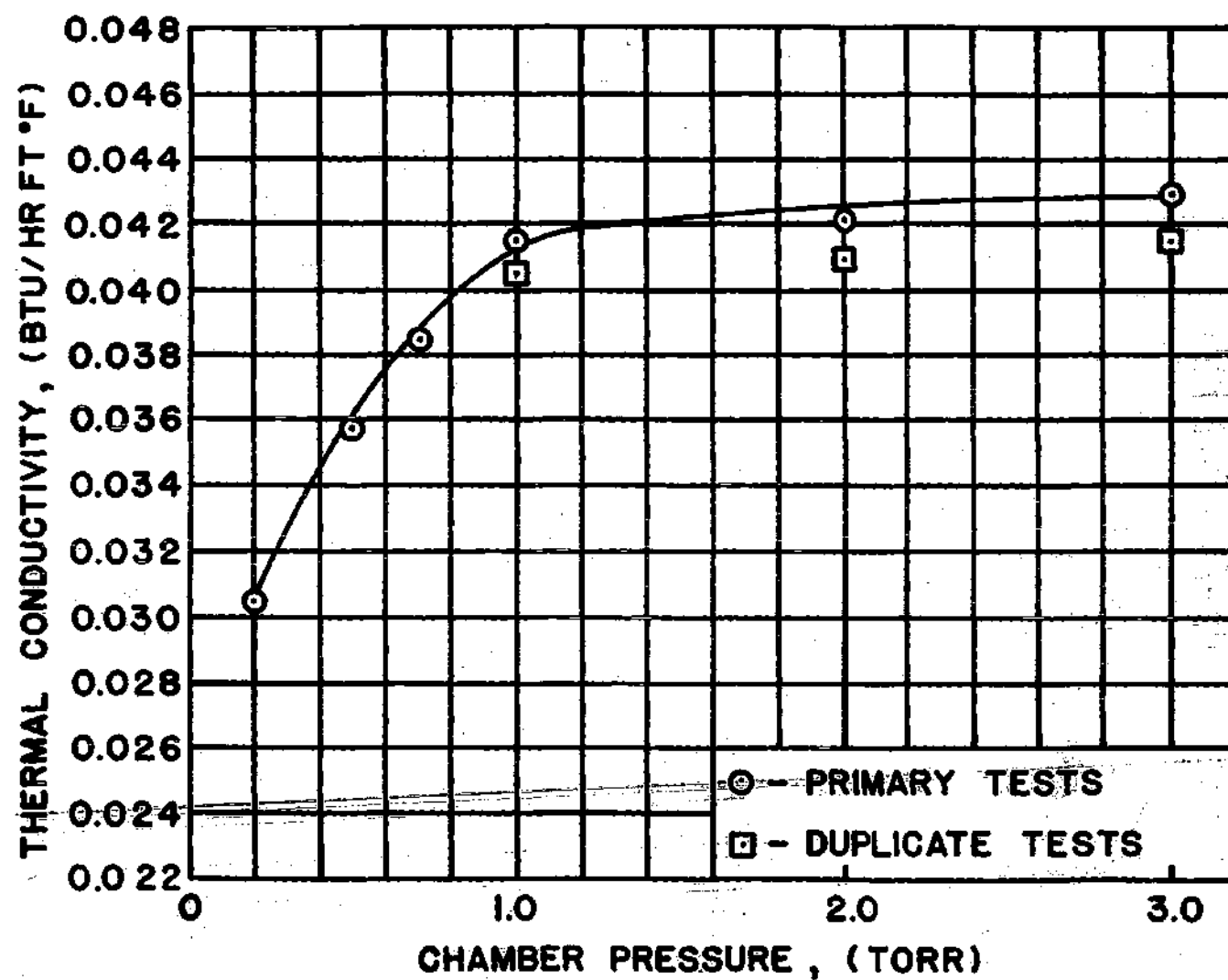


Figure 8. Thermal Conductivity of Freeze-Dried Beef versus Total Chamber Pressure (Heat of Sublimation = 1220 Btu/lb_m).

in equation 5.2 could not be accurately measured and as a result this value of the conductivity is not considered to be accurate.

Table 2. Surface and Interface Temperatures for Conductivity Tests

Chamber Pressure torr	Average Surface Temp. °F	Average Interface Temp. °F
0.2	102	-30
0.5	101	-14
0.7	100	-8
1.0	100	0
2.0	100	13
3.0	100	22.5
4.0	100	28.5

At the lowest chamber pressure, 0.2 torr, the recorded interface temperature was -30°F insuring that the product was solidly frozen. In some tests made at 0.2 torr, the interface temperature was at -38°F. As a point of interest, it was observed in all tests that at the beginning of each run the temperature in the frozen product was considerably below the value recorded in Table 2 and then rose to the listed equilibrium value for most of the run. For instance at 0.2 torr the center temperature fell as low as -53°F at the beginning of one test. The interface temperature was quite sensitive to small changes in chamber pressure especially at lower chamber pressures thus requiring close control of

the pressure to maintain equilibrium.

One of the tests made at a chamber pressure of 1 torr yielded a conductivity of 0.035 Btu/hr ft²F (for $\Delta h = 1220$ Btu/lb_m) which is considerably lower than the others recorded at the same pressure (see Figure 8). Upon inspection of the sample at the conclusion of the test, it was discovered that the direction of the fibers of the beef was at an angle of nearly 30° to the normal to the surface. This illustrated the directional dependence of the thermal conductivity of freeze-dried beef and emphasizes the importance of allowing the heat transfer to take place parallel to the grain of the beef.

Table 1 also has listed the conductivity values where the convective contribution to heat transfer has been neglected. The error involved in neglecting this contribution is in the order of 2.5 to 3.5 per cent.

Figure 9 compares the results obtained by Harper, et al. (4) for various gases in the pores of the dried beef and the results of this work using the heat of sublimation for pure ice. As was mentioned in Chapter I, the results of this work might have been expected to correlate with Harper's results for neon on the basis of the molecular weights of neon and water being almost equal. However, it is easily seen that this is not the case and that the quasi-steady results of this work are on the average 25 to 30 per cent higher than the data for neon. The values obtained by Harper for the lighter weight gases such as helium and hydrogen compare fairly well at certain pressures with the results of this work but there is no apparent basis for comparison in these cases. The conductivity obtained by Rolfe (5), mentioned in Chapter I, at 0.17 torr of 0.033 Btu/hr ft²F using the Δh of pure ice agrees fairly well

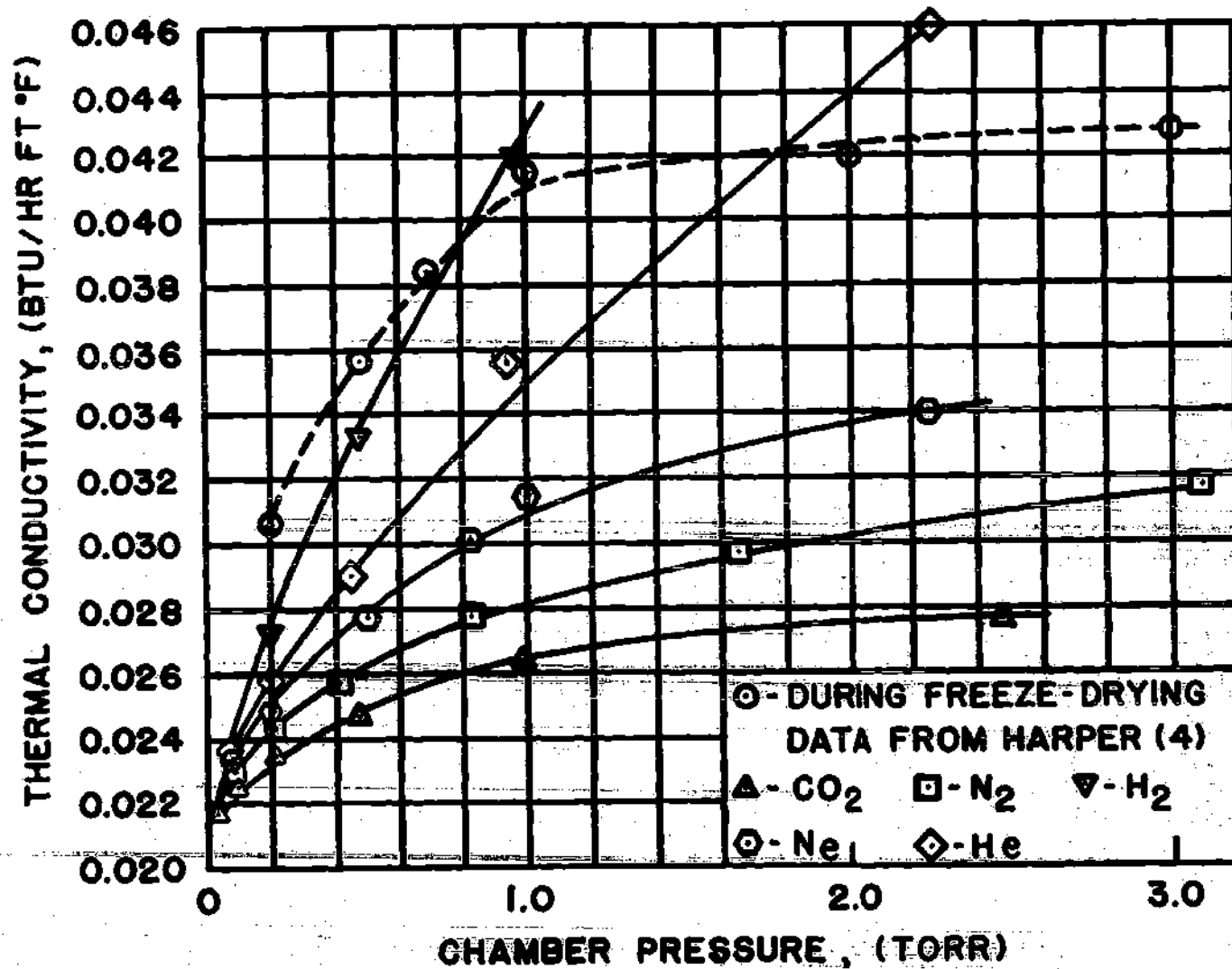


Figure 9. Comparison of Thermal Conductivity Results During Freeze-Drying and Steady-State Thermal Conductivities.

with the results of this work obtained at 0.2 torr also for the same Δh . Note that for all the gases used by Harper the conductivity reaches a constant value at 0.02 torr and lower. At these pressures, the gas does not contribute to conducting thermal energy and thus all heat conduction is due to the solid meat structure. Presumably the conductivity measured under freeze-drying conditions would have approached the value Harper records at 0.02 torr. However, the existing equipment did not permit testing below 0.2 torr. At this writing, the apparent discrepancy between the data obtained under quasi-steady conditions and that from steady-state conditions has not been explained satisfactorily.

Results for Alternate Conductivity Test

As was mentioned in Chapter II, an alternate method to measure the thermal conductivity of freeze-dried beef was used on completely dried beef samples. The procedure consisted of placing a piece of pure ice between two previously dried beef samples and then supplying heat to the outside surface of the samples. This allowed the ice to sublime and the resulting vapor passed through the beef samples into the chamber. In this way data points were obtained over short time intervals as there was a slight decrease in flow rate. The chamber pressure used in this test was 1 torr. It was necessary to maintain careful control to avoid overheating the meat surface. Based upon equation 2.10 the thermal conductivity of the sample could be determined for this arrangement. The sample lengths, L , were 0.24 inches and the diameters were 2.9 inches. The heat of sublimation, Δh , used was 1220 Btu/lb_m. The average conductivity value obtained for this test was 0.043 Btu/hr ft²F. Of course

the heat of sublimation for this test was known exactly since a piece of pure ice was used. We thus have three values of the thermal conductivity of freeze-dried beef at a chamber pressure of 1 torr. These are shown in Table 3 below.

Two general interpretations can be made from the results shown in Table 3. The first comes from a comparison of the results of the ice sample test and the results which were obtained under drying conditions where k was calculated using $\Delta h = 1220 \text{ Btu/lb}_m$. The relatively

Table 3. Thermal Conductivities at
1 torr Chamber Pressure

Chamber Pressure torr	k_i $\frac{\text{Btu}}{\text{hr ft } ^\circ\text{F}}$	k^* $\frac{\text{Btu}}{\text{hr ft } ^\circ\text{F}}$	k^{**} $\frac{\text{Btu}}{\text{hr ft } ^\circ\text{F}}$
1.0	0.043	0.0416	0.0503

where:

k_i - Conductivity from special ice test

k^* - Conductivity, k , calculated using $\Delta h = 1220 \text{ Btu/lb}_m$

k^{**} - Conductivity, k , calculated using $\Delta h = 1488 \text{ Btu/lb}_m$

good agreement between these two tests appears at first to indicate that the thermal properties of ice and frozen beef juices are very nearly equal. In particular, the heats of sublimation of frozen beef juices and pure ice are nearly equal. The second interpretation, however, comes from a comparison of the results of the ice sample test and the results obtained under drying conditions where k was calculated using $\Delta h = 1488 \text{ Btu/lb}_m$. If the heat of sublimation of frozen beef juices is correct, the

difference between the conductivities of these two tests may simply be the difference between the conductivities of beef juice-vapor and water-vapor. In this case the apparent agreement mentioned in the first case would simply be a coincidence. However, at present there is no satisfactory explanation of these results and further studies are needed.

The relative difficulties inherent in the test with pure ice prevents complete confidence in the results obtained in Table 3. As mentioned above the dried meat samples were approximately 0.24 inches in diameter. Perfectly symmetrical samples of this size are very difficult to make and heat transfer calculations are seriously affected by any error in measurement on this scale. Also, the test sample, which included two dry pieces of beef with thermocouples on each surface and a piece of pure ice between them, was difficult to assemble. However, with some refinements, this test appears to be an excellent technique to use for comparison with the results obtained by Harper, et al. (4) under steady-state conditions. Its value for determining the conductivity of freeze-dried beef for use in freeze-drying calculations may be questionable as it still does not represent the true situation.

Calculated Drying Times

The conductivity values obtained in this work were used in the analytical equations of Dyer, et al. (3) to calculate drying times. The equations used and a brief derivation are presented in Appendix B. The analysis is based on a quasi-steady solution and requires numerical techniques for solution. According to Dyer (10) this solution is accurate to within two per cent of the exact solution also derived by him and

is easier to apply. It was assumed that one surface was at 100°F, the other was adiabatic and the thickness was 0.25 inches. This model corresponds to the drying that occurs from one side of the experimental arrangement considering each surface at 100°F and the center plane as adiabatic. Total chamber pressures of 1.0, 2.0 and 3.0 torr were considered with water-vapor partial pressures of 0.9, 1.4 and 2.3 torr which were measured in the experimental work. Experimental values for the diffusion coefficient obtained by Dyer (10) were used. These were 0.017, 0.013, 0.011 ft²/sec corresponding to the chamber pressures of 1, 2 and 3 torr respectively. For the permeability, values of 0.00112, 0.00940 and 0.00085 mole ft/lb_f hr were used also obtained by Dyer at pressures of 1, 2 and 3 torr.

Table 4. Comparison of Predicted Drying Times
With Experimental Drying Times

Chamber Pressure torr	t_p^* hrs	t_p^{**} hrs	t_o hrs	t_p^* Neon hrs
1.0	3.51	3.51	3.85	4.62
2.0	3.76	3.79	3.75	4.81
3.0	4.05	4.06	4.20	4.96

where:

t_p^* - Predicted drying time calculated using
 $\Delta h = 1220 \text{ Btu/lb}_m$.

t_p^{**} - Predicted drying time calculated using
 $\Delta h = 1488 \text{ Btu/lb}_m$.

t_o - Observed experimental drying time.

The drying times were calculated with the aid of a digital computer and are listed in Table 4 along with the observed experimental drying times. Both sets of conductivity data listed in Table 1 were used to calculate drying times. Of course it is necessary to use the same heat of sublimation in the drying time equations that was used to calculate the conductivity value. As can be seen, there is no significant difference between the calculated drying times using either set of conductivities and the corresponding heat of sublimation. This result is attributed to an approximately linear relationship between the thermal conductivity and the heat of sublimation illustrated in equation 2.7 and the drying time equation B.16. Thus an increase in Δh results in a corresponding increase in k . The experimentally observed drying times are also listed in Table 4. These times are derived from Figures 10 through 12 of the specific moisture loss versus time. The theoretical results show that the drying times increase with increasing chamber pressure. The experimental results appear at first to contradict this trend. However, the smaller drying time observed at a chamber pressure of 2 torr as opposed to that at 1 torr is explained by the fact that the sample used at 2 torr was approximately 4 grams lighter than the one used at 1 torr. This appears to be a small difference at first glance but when one considers the small samples used this difference becomes quite significant. Figure 12 presents two curves for the chamber pressure of 3 torr. The faster drying time for one of the samples resulted from its slightly lighter weight and more importantly the higher surface temperature at which it was dried.

Due to the similarity between the equations derived in Chapter II

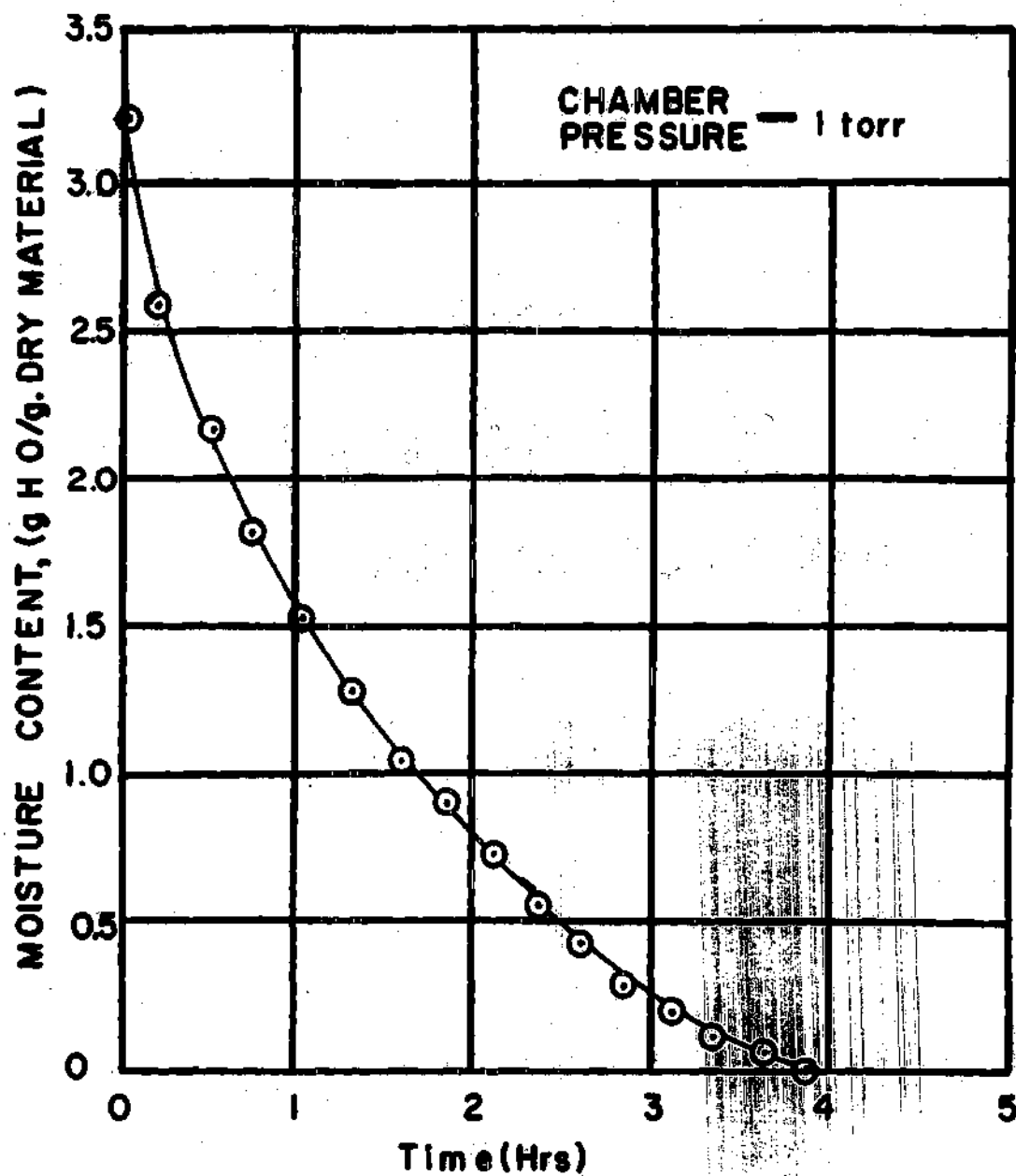


Figure 10. Moisture Content versus Time, 1 torr.

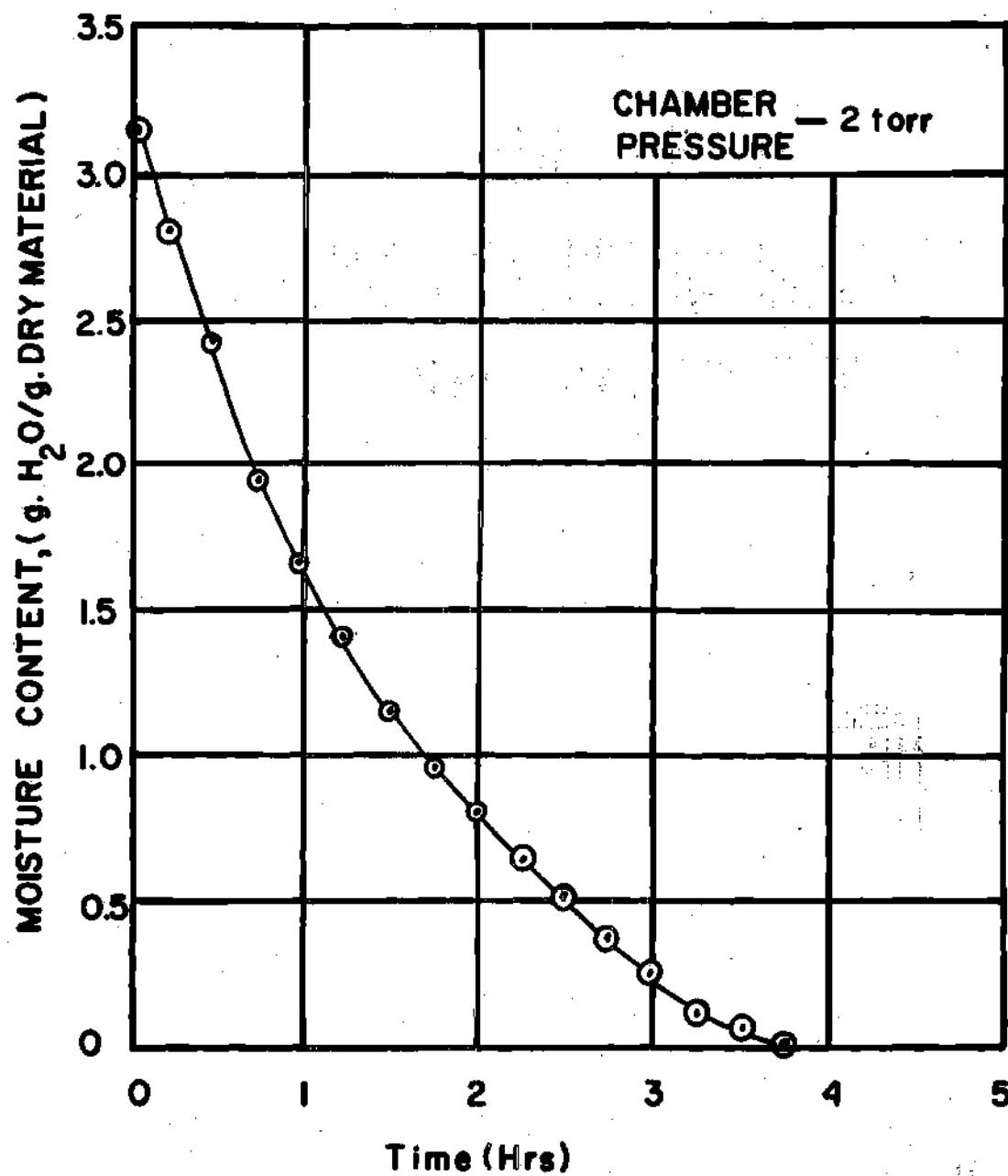


Figure 11. Moisture Content versus Time, 2 torr.

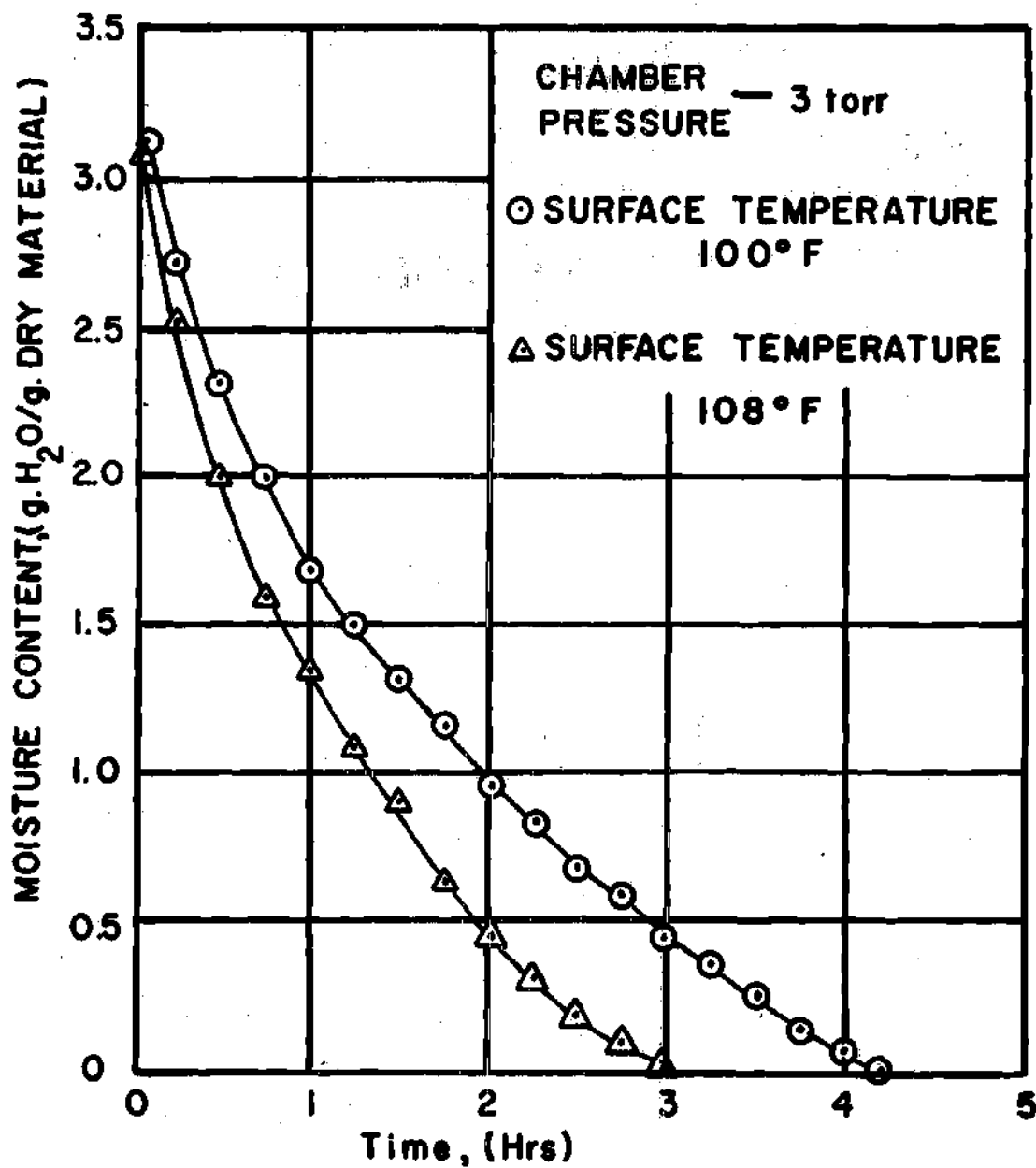


Figure 12. Moisture Content versus Time, 3 torr.

and those in Appendix B, it would be expected that calculated drying times would compare well with the observed drying times of the experiments from which the conductivity data were obtained. For, in one case we treat the conductivity, k , in equation 2.7 as unknown and calculate it from experimental measurements of the flow rate, R . However, in equation B.8 we treat the conductivity as known and calculate the flow rates. Thus the only purpose served is to check for computational errors in calculating the conductivity. To check for the true accuracy of the conductivity data, the results should be used to compare theoretical drying rates with independently observed drying rates. Dyer and Sunderland (12) have used the conductivity data of this work in some more recent theoretical equations for sublimation dehydration rates for comparison with experimental results obtained by Hatcher (9). Hatcher freeze-dried beef at several pressures by heating the sample to 100°F on one face and attempting to thermally insulate the opposite face. Thus his experimental conditions correspond to the assumed conditions mentioned at the beginning of this section.

Hatcher's data and the theoretical results are plotted in Figures 13 and 14 for chamber pressures of 1 and 2 torr showing the interface position as a function of time. It can be seen that the analytical and experimental results correspond closely during the initial stages of drying. However, as drying proceeds, the experimental data indicate a progressively faster drying rate than the theoretical results. This observation can be explained by a careful analysis of Hatcher's experiments. Initially during the experiments no temperature gradient or heat transfer existed in the frozen region. At later stages of drying, a

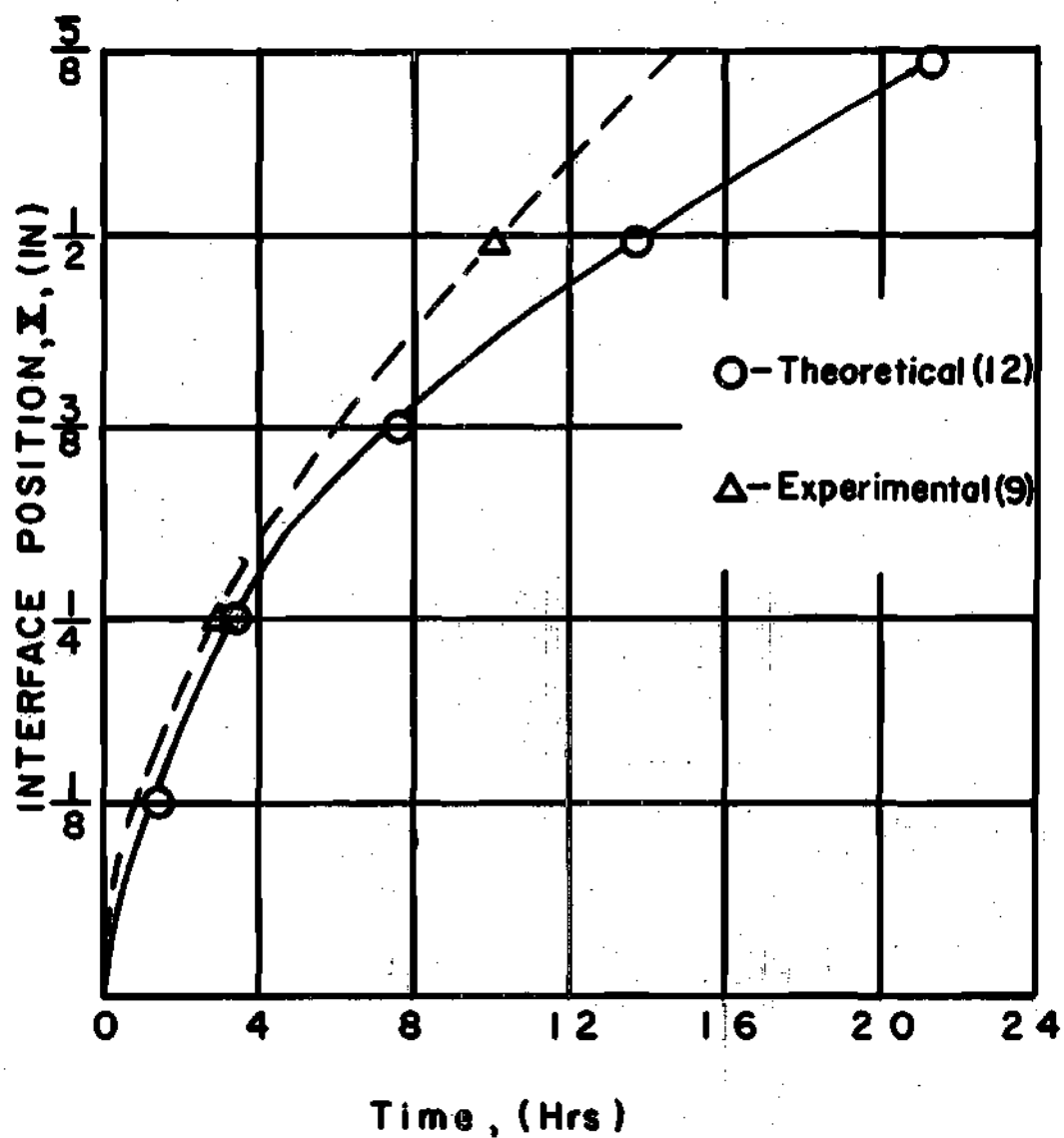


Figure 13. Interface Position versus Time, 1 torr.

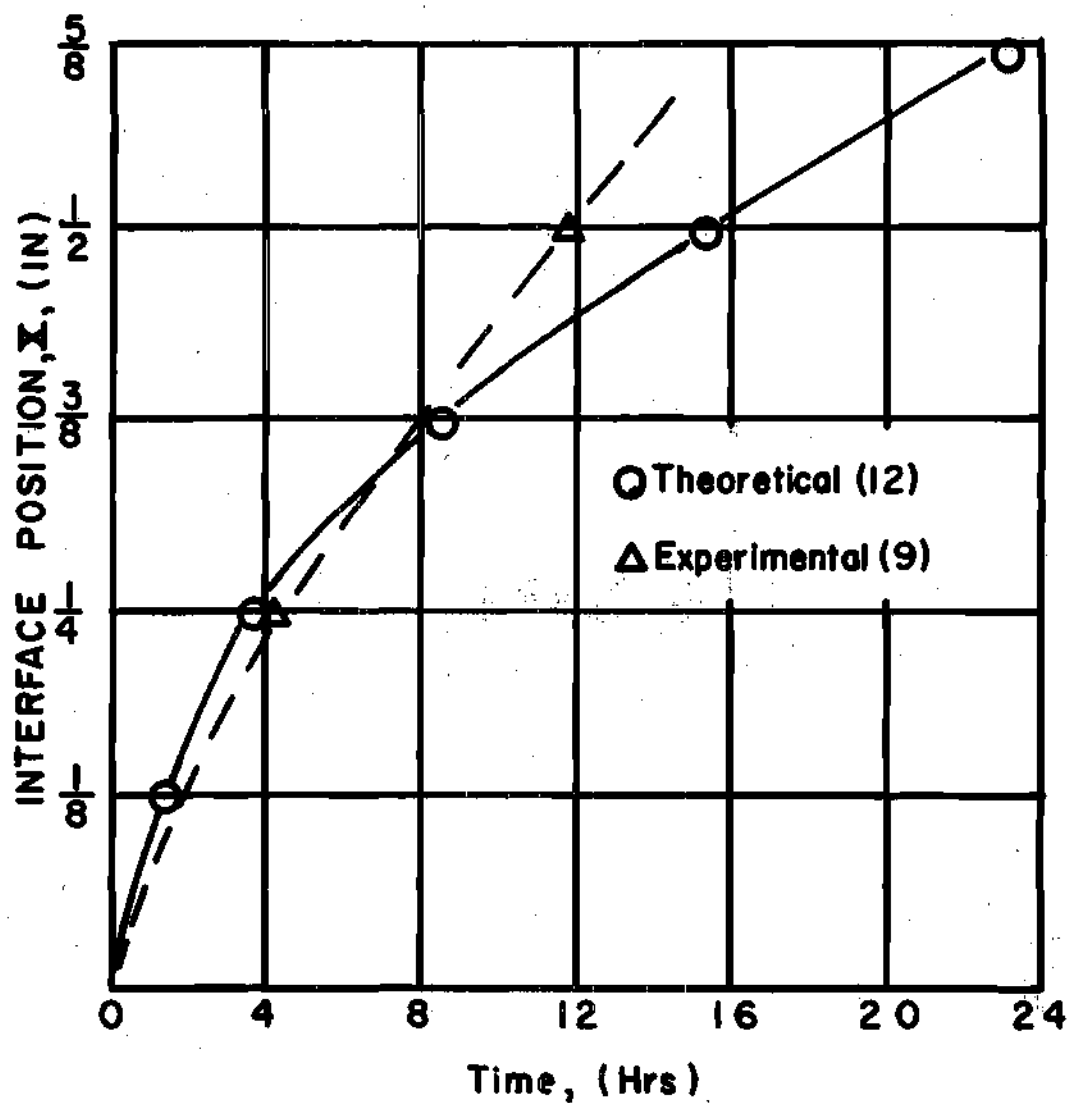


Figure 14. Interface Position versus Time, 2 torr.

temperature difference of 2 or 3°F resulted from imperfect thermal insulation. Hill (13) showed the thermal conductivity of the frozen beef is 20 times the conductivity of dried beef. Hence the heat conducted through the frozen layer approaches the same magnitude as that through the dried layer. Indeed, proportionately, more heat is transferred through the frozen layer as drying proceeds and the conduction path through the frozen region becomes shorter.

Thus it is seen from these observations that during the early stages of drying the theoretical boundary conditions are nearly satisfied. As time increases, the progressive divergence between theory and experiment can be ascribed to the proportionately larger amounts of heat transfer through the frozen layer resulting in an increased sublimation rate in the experimental case. Thus, it is concluded that the conductivity data obtained under the quasi-steady method allows accurate predictions of sublimation drying rates using analytical equations derived for this purpose. Also, there is little difference observed in the predicted drying rates, using either the Δh of pure ice or the Δh of frozen beef juices provided the proper conductivity value associated with each Δh is used.

In Table 4 drying times are calculated using values for the thermal conductivity of 0.031, 0.033, and 0.035 Btu/hr ft°F obtained by Harper, et al. (4) for neon gas. The predicted drying times using the data for neon gas were calculated using the heat of sublimation for pure ice. Had the drying times been calculated using the higher heat of sublimation they would have been even further from the observed drying times. As it turned out, the drying times listed in Table 4, using the conduc-

tivities for neon gas, are from 25 to 30 per cent longer than the drying times calculated using the data obtained under quasi-steady conditions.

Partial Pressures

As mentioned in Chapter III, an electronic element sensitive to water-vapor partial pressure was installed in the vacuum chamber. The read out was converted to relative humidity on a graph supplied by the manufacturer. Then using steam tables the partial pressure of the water-vapor was easily calculated. Mayne (14) has shown that the sensing element accurately measures the partial pressure of the water-vapor in the drying chamber at total pressures of 1, 2 and 3 torr. However, at pressures below 1 torr the element was not sensitive enough to accurately measure the partial pressure of the water-vapor. Although the partial pressure was measured at the exit of the vacuum chamber (see Figure 3), the gradient in partial pressure between there and the surface of the meat is so small that it is neglected. Thus it is assumed that the water-vapor partial pressure at the meat surface is equal to the experimentally measured value. The measured values of the water-vapor partial pressure were used in calculating the drying times mentioned above. The equations also predict the interface temperature which is of course a function of the partial pressure at the interface (see Appendix B, equation B.10). These predicted values are listed in Table 5 along with the experimentally observed interface temperatures. As can be seen, the agreement is very good between theory and experiment.

The results outlined above confirm the accuracy of the data obtained under quasi-steady conditions and indicate the necessity of measuring thermal conductivities of freeze-dried foods using this technique.

Table 5. Comparison of Predicted Interface Temperatures and Experimental Interface Temperatures

Chamber Pressure torr	Water-vapor Partial Pressure, torr	T_i Predicted °F	T_i Observed °F
1.0	0.9	5.51	5.0
1.0	0.7	2.56	0.0
2.0	1.4	14.7	13.0
3.0	2.32	22.1	22.5

Experimental Errors

In this work, two sources of error were present. One was excess drying around the edge of the sample which is not consistent with the one-dimensional model and can significantly affect the value of the conductivity. The other was the thermal conduction error associated with the thermocouples which can also produce serious errors.

Two steps are necessary to insure that one-dimensional heat transfer occurs through the dry layer of the beef sample. One is that the surface area to edge area ratio be as large as possible which is perhaps the most important requirement. For this work, the diameter of the samples was three inches as this was the largest homogeneous sample which could be obtained. The thickness of the samples was approximately a half inch which was thick enough to insure sufficient drying time to gather data, but thin enough to insure a uniform heat flux through the dry layer. Figures 15 and 16 are cross-sections of partially dried samples illustrating the effects of surface area to edge area on the amount

of end drying that occurs during freeze-drying. Figure 15 for a half inch thick sample shows that very little end drying has occurred even in the advanced stages of the process. Figure 16 for a one and a half inch thick sample shows that a considerable amount of end drying has occurred with quite a bit of drying time remaining. In a test made at 1 torr on a one and a half inch sample, the calculated conductivity was 0.057 Btu/hr ft²F which is considerably higher than the value of 0.0416 Btu/hr ft²F recorded on the half inch samples and illustrates the importance of preserving the one-dimensional model as nearly as possible. The second item to insure one-dimensional heat transfer was an abundance of insulation surrounding the edge of the sample to reduce the radial heat flux as much as possible. The arrangement used in this work is described in Chapter IV.

The thermocouple conduction error may cause serious problems in conductivity measurements. The thermocouples attached to the surfaces of the sample are subject to large amounts of radiation flux from the heaters. If they are slightly oxidized or if they have a dirt film, they may absorb a considerable amount of thermal radiation and being attached to the surface of a material of low thermal conductivity they cannot efficiently dissipate the thermal energy which is stored in the wire. As a result, considerably higher temperatures may be recorded at the thermocouple junction than are present over the surface of the sample. Before corrective measures were taken, errors in excess of 20°F were noted. The most practical solution to this problem was to use very small gage thermocouple wire (36 gage) and to embed the wire just under the surface of the meat. Although the temperature gradient is large in

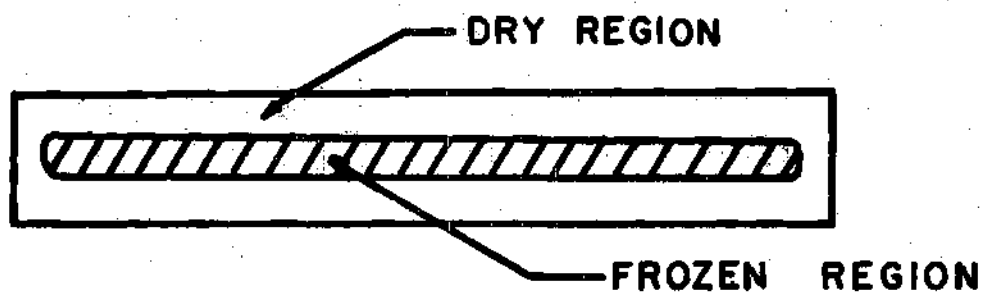


Figure 15. Cross-section of a Partially Dried Beef Sample, 1/2-inch Thick.

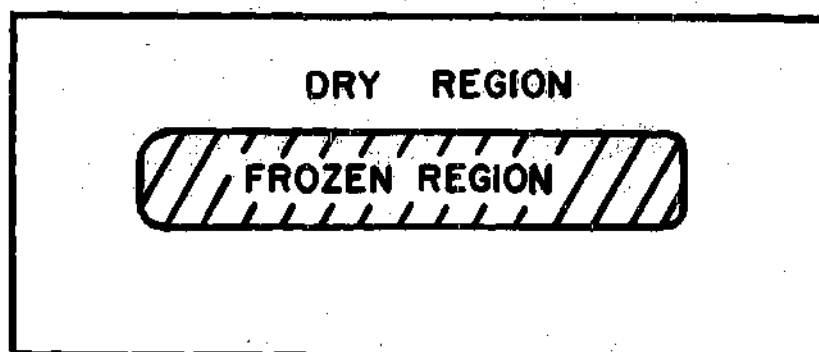


Figure 16. Cross-section of a Partially Dried Beef Sample, 1-1/2 inches Thick.

the dry layer, this procedure gave the most reliable and consistent results. Conduction errors to the center thermocouple were virtually eliminated by inserting the thermocouple radially into the center and then wrapping it once around the edge of the sample before it passed out through the insulation and into the atmosphere of the chamber.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

The major conclusions to be drawn from the experimental and theoretical results of this work are:

1. The thermal conductivity of freeze-dried beef decreases with decreasing chamber pressure due to the decreased conductivity of the gas in the void spaces.
2. At a chamber pressure of 4 torr, the beef sample thawed thus making this pressure unsuitable for conventional freeze-drying. At chamber pressures of 3 torr and below, the sample remained frozen allowing sublimation dehydration to take place.
3. The thermal conductivity of freeze-dried beef is directionally dependent with the maximum value occurring parallel to the fiber direction.
4. The sensible heat gain to the vapor flowing through the dry layer during freeze-drying is in the order of 3 per cent of the total heat required for sublimation of the ice phase.
5. The conductivities measured under freeze-drying conditions do not seem to be compatible with those measured on dry samples by Harper, et al. (4) under steady-state conditions in the presence of various gases. It is thought that this may be the result of the peculiar nature of the beef juice-vapor causing it to have a high thermal conductivity. Also, it may be that there is some reaction between the vapor

stream in the dry layer and the solid beef matrix causing the higher conductivity. However, at present there is no satisfactory explanation for the difference.

6. The thermal conductivity may be determined by subliming a piece of pure ice between two dry beef samples. This approximates steady-state procedures but may not give the true conductivity under freeze-drying conditions due to thermal differences between water-vapor and beef juice-vapor.

7. The conductivities measured under freeze-drying conditions calculated using the higher heat of sublimation reported by Dyer et al. (11) are about 22 per cent greater than those calculated using the heat of sublimation of pure ice.

8. Calculated drying times compare very well with experimentally observed drying times using conductivities calculated with either heat of sublimation. Both experimental and theoretical drying rates show that drying is faster for lower chamber pressures.

The following items are presented as a logical extension of the work which has been presented in this thesis:

1. Work should be done to explain the difference between conductivities measured under freeze-drying conditions and under steady-state conditions.

2. Although it is thought that the heat of sublimation for frozen beef juices is very accurate, it should be confirmed through further experimentation so that conductivities measured under freeze-drying conditions may be reported as accurately as possible.

APPENDIX A

TABULAR DATA FOR CONDUCTIVITY TESTS

Table 6. Weight Loss for Test Runs at Each Chamber Pressure

Time hrs	Weight Loss, grams					
	0.2 torr	0.5 torr	0.7 torr	1.0 torr	2.0 torr	3.0 torr
0	0	0	0	0	0	0
.25	0.4	0.4	1.6	1.7	1.2	1.4
.50	3.1	4.3	3.2	3.8	4.5	5.2
.75	6.2	10.2	6.9	8.7	9.6	10.3
1.00	11.0	15.9	11.9	14.3	15.7	15.3
1.25	16.1	20.4	17.5	19.5	19.8	18.9
1.50	20.5	24.0	22.0	23.1	23.1	21.8
1.75	24.0	27.1	25.3	26.1	25.7	24.3
2.00	26.8	29.5	28.3	28.8	28.3	26.7
2.25	29.4	32.2	31.1	31.3	30.6	28.8
2.50	31.9	34.3	33.2	33.8	32.6	30.7
2.75	34.2	36.2	35.4	36.1	34.5	32.4
3.00	36.2	37.5	37.4	38.1	36.0	34.2
3.25	38.2	*	39.2	39.6	37.6	35.7
3.50	40.0		41.4	41.2	39.8	
3.75	41.3			42.8	40.0	
4.00	42.9			44.0	*	
	*			*		
Initial Weight	55.9	49.6	55.9	57.7	53.9	55.0

* Completely Dry

APPENDIX B

DERIVATION OF DRYING TIME EQUATION

The analytical results of this section are derived in a thesis by Dyer (10) and the expressions developed are for the case where heat is supplied to one surface of the sample and all the other sides including the opposite surface are adiabatic.

The equation of motion for the water-vapor species which also serves as the definition of the average diffusion coefficient is,

$$N_w X = \rho D_e \ln \frac{1 - y_{w0}}{1 - y_{wX}} \quad (B.1)$$

We now seek expressions for the terms $N_w X$, y_{w0} and y_{wX} .

The differential equation describing the temperature distribution in the dry layer is

$$\frac{d^2 \theta}{dx^2} - N_w \frac{\bar{c}_p}{k} \frac{d\theta}{dx} = 0 \quad (B.2)$$

where θ is a non-dimensional temperature defined by

$$\theta = \frac{T_x - T_0}{T_X - T_0} \quad (B.3)$$

The boundary conditions associated with Equation B.2 are

$$\begin{aligned} \theta &= 0 \quad \text{at} \quad x = 0 \\ \theta &= 1 \quad \text{at} \quad x = 1 \end{aligned} \quad (B.4)$$

An energy balance at the interface position gives the following

$$(T_0 - T_X) k \left. \frac{d\theta}{dx} \right|_{x=0} - \tilde{c}_p N_w (T_0 - T_X) = N_w \Delta \tilde{H} \quad (\text{B.5})$$

The solution to equation B.2 with the boundary conditions gives

$$\theta = \frac{1 - \exp(-N_w \tilde{c}_p x/k)}{1 - \exp(-N_w \tilde{c}_p X/k)} \quad (\text{B.6})$$

Note that with the assumption of a small flow rate, N_w , we may expand the exponential terms in Taylor series and obtain

$$\theta \approx \frac{1 - 1 + N_w \tilde{c}_p x/k}{1 - 1 + N_w \tilde{c}_p X/k} = \frac{x}{X} \quad (\text{B.7})$$

Thus the temperature distribution is essentially linear. This bears out the assumption of a linear temperature gradient used in deriving equation 2.7 on page 16. Substituting B.7 into equation B.5 yields

$$N_w X = \frac{-k(T_X - T_0)}{-\Delta \tilde{H} + \tilde{c}_p (T_0 - T_X)} \quad (\text{B.8})$$

The equation of motion for the gas mixture is

$$P_X - P_0 = \frac{N_w X}{z} \quad (\text{B.9})$$

Also an empirical expression for the water-vapor partial pressure at the interface is

$$P_{wX} = \exp (27.7 - 12900/T_X) \quad (B.10)$$

Then employing the definition of the molar concentration,

$$Y_{wX} = \frac{P_{wX}}{P_X} \quad (B.11)$$

the following expression is obtained.

$$Y_{wX} = \frac{\exp (27.70 - 12,900/T_X)}{P_O + N_w X / \tilde{e}} \quad (B.12)$$

The molar concentration at the surface of the sample is defined by an equation similar to equation (B.11), thus

$$Y_{wO} = \frac{P_{wO}}{P_O} \quad (B.13)$$

Substituting equations B.8, B.12, and B.13 into B.1 gives

$$\frac{k(T_O - T_X)}{-\Delta\tilde{H} + \tilde{c}_p(T_O - T_X)} = \rho D_e \ln \frac{1 - P_{wO}/P_O}{1 - \left[\frac{\exp(27.7 - 12,900/T_X)}{P_O + \frac{k(T_O - T_X)}{\tilde{e}[-\Delta\tilde{H} + \tilde{c}_p(T_O - T_X)]}} \right]} \quad (B.14)$$

By trial and error this equation may be used to calculate the temperature at the interface. Note that in equation B.8 the magnitude of $N_w X$ is a constant. The relationship between the flow rate and interface velocity is

$$N_w = \rho_i \sigma \frac{dX}{dt} \quad (B.15)$$

Substituting for N_w from equation B.8 into equation B.15, integrating and noting that at $t = 0$, $X = 0$ gives

$$t = \frac{\rho_1 \sigma X^2}{2 \left[\frac{k(T_0 - T_X)}{-\Delta\tilde{H} + \tilde{c}_p(T_0 - T_X)} \right]} \quad (\text{B.16})$$

Thus the time to dry to any position X may be easily calculated. Calculations of interface temperature and drying time using equations B.14 and B.16 are presented in Chapter V.

APPENDIX C

EQUIPMENT AND INSTRUMENT SPECIFICATIONS

Electric Heaters

Manufacturer.....Watlow Electric Company
Type.....11" x 15" flat plate
Capacity.....1700 watts

Vacuum Pump

Manufacturer.....W. M. Welch Scientific Co.
Model.....1397 B
Capacity.....375 liters/min. of free air

Weighing Balance

Manufacturer.....Mettler Balances Inst. Co.
Model.....K5T
Capacity.....0 - 2000 grams
Minor Division.....1 gram

Absolute Pressure Gage

Manufacturer.....Wallace and Tiernan Co.
Model.....FA-160150
Range.....0.1 to 20 torr
Minor Division.....0.1 torr
Accuracy.....0.33% of full scale range

Humidity Transducer

Manufacturer.....Hydrodynamics, Inc.
Model.....Class A 4-4812 White
Range.....1.5 to 8.6% RH
Accuracy..... $\pm 1.5\%$ RH

Humidity Transducer Indicator

Manufacturer.....Hydrodynamics, Inc.
Model.....15-3000

LITERATURE CITED

1. Harper, J. C. and A. L. Tappel, Advances in Food Research, Vol. VII, Academic Press, New York, 1957, pp. 171-234.
2. Harper, J. C., C. O. Chichester, and T. E. Roberts, "Freeze-Drying of Foods," Agricultural Engineering, Vol. 43, No. 2, 1962, pp. 78-81.
3. Dyer, D. F., and J. E. Sunderland, "Heat and Mass Transfer Mechanisms in Sublimation Dehydration," Progress Report for Public Health Service Research Grant EF 00102-02 from the Division of Environmental Food Protection, 1965.
4. Harper, J. C. and Ahmed F. Sahrighi, "Thermal Conductivities of Gas Filled Porous Solids," I and EC Fundamentals, Vol. 3, No. 4, 1964, pp. 318-324.
5. Rolfe, E., "The Freeze-Drying of Fish and Meat," Freeze-Drying of Foodstuffs, Cotson, S., and Smith, D. B., eds., Columbia Press, Manchester, England, 1963, pp. 119-131.
6. Lusk, G., M. Karel, and S. A. Goldblith, "Thermal Conductivity of Some Freeze-Dried Fish," Food Technology, Vol. 18, No. 10, 1964, pp. 121-124.
7. Meffert, H. F. Th., Institute for Research on Storage and Processing of Horticultural Produce, Wageningen, The Netherlands, Personal Communication, 1965.
8. Schneider, P. J., Conduction Heat Transfer, Addison Wesley, New York, 1956, pp. 218-221.
9. Hatcher, J. D., The Use of Gamma Radiation to Measure Moisture Distribution During Drying Processes, Master's Thesis, Georgia Institute of Technology, Atlanta, Georgia, 1964.
10. Dyer, D. F., Transport Phenomena in Sublimation Dehydration, Ph.D. Thesis, Georgia Institute of Technology, Atlanta, Georgia, 1965.
11. Dyer, D. F., D. K. Carpenter, and J. E. Sunderland, "Equilibrium Vapor Pressure of Frozen Bovine Muscle," Accepted for publication in Journal of Food Sciences.
12. Dyer, D. F., and J. E. Sunderland, "The Transient Temperature Distribution during Sublimation Dehydration," Progress Report for Public Health Service Research Grant EF 00102-02 from the Division of Environmental Food Protection, 1966.

13. Hill, J. E., The Thermal Conductivity of Beef, Master's Thesis, Georgia Institute of Technology, Atlanta, Georgia, 1966.
14. Mayne, R. W., Humidity Measurement under Low Vacuum Conditions, Master's Thesis, Georgia Institute of Technology, Atlanta, Georgia, 1965.